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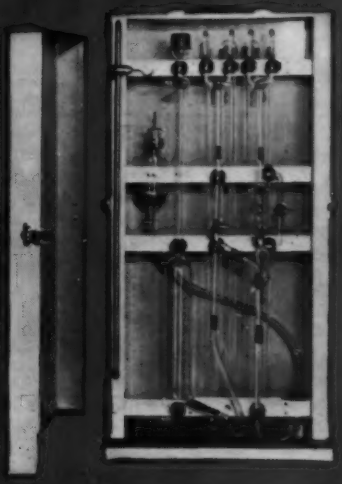
LABORATORY PRACTICE

889

RESEARCH · CONTROL · TEACHING
VOL. 10 No. 12 Published Monthly DECEMBER 1961

gas chromatography by the Janák method



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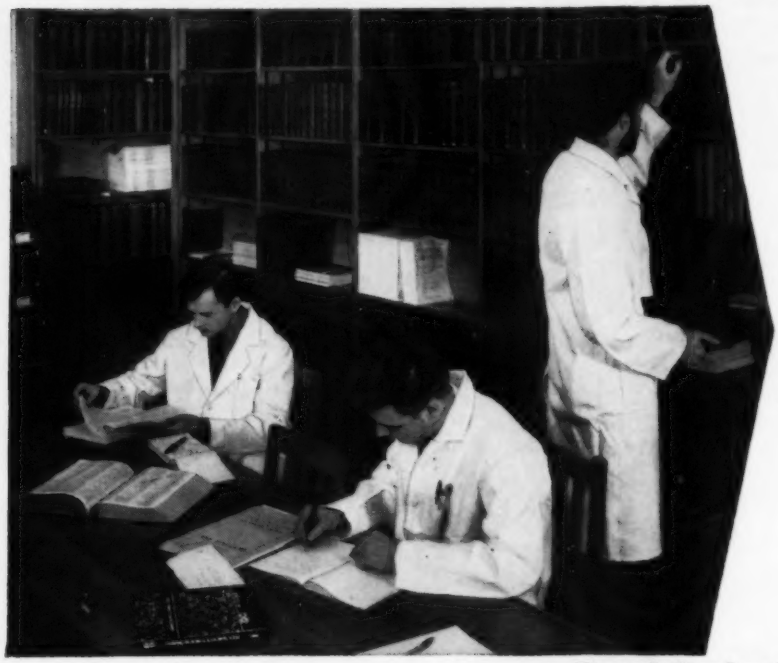

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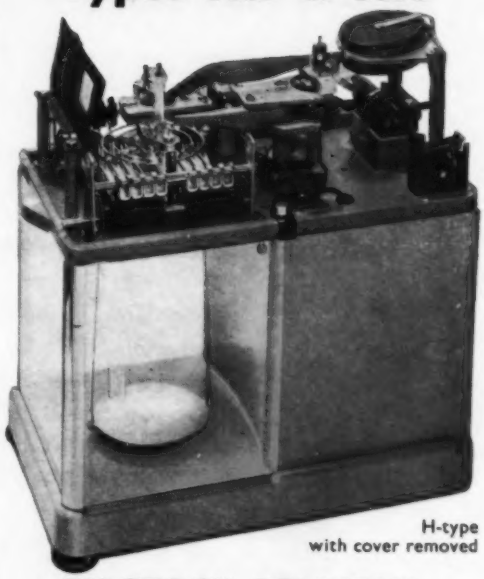

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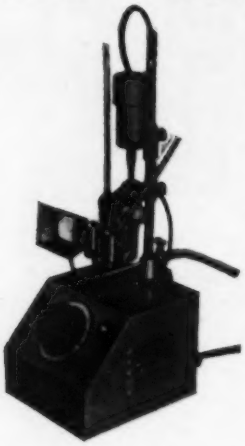

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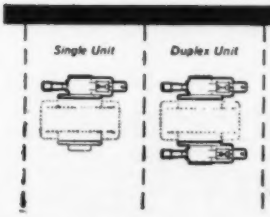

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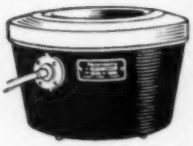

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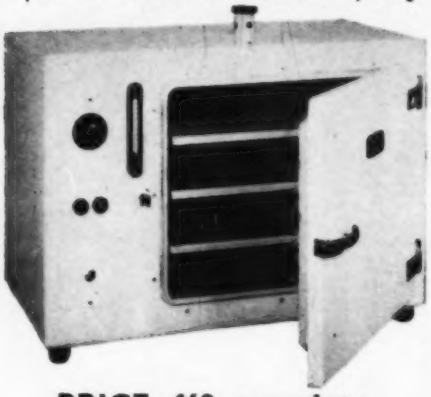

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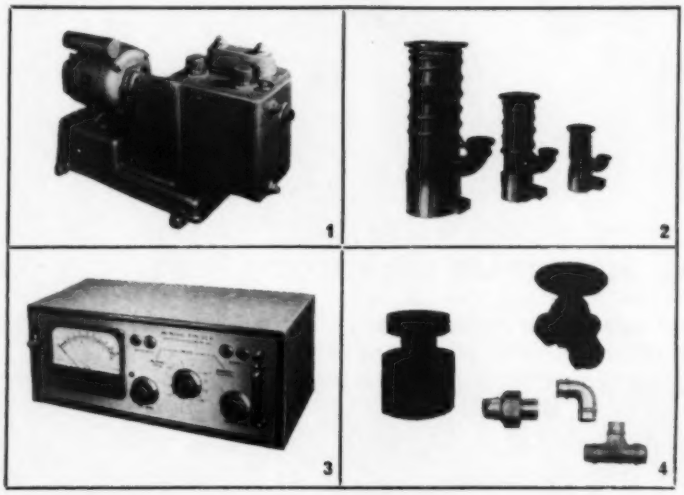

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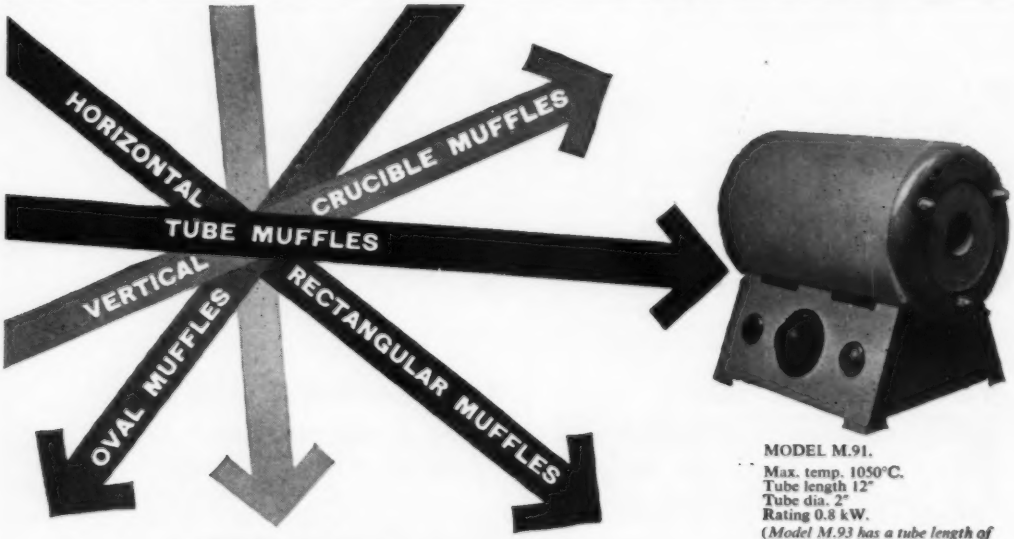

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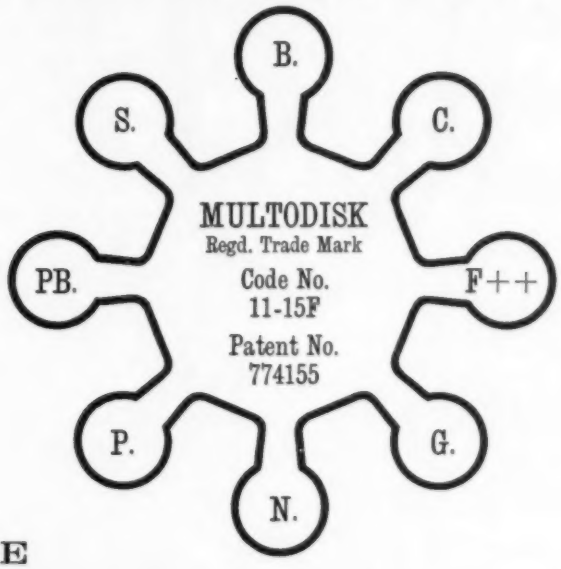

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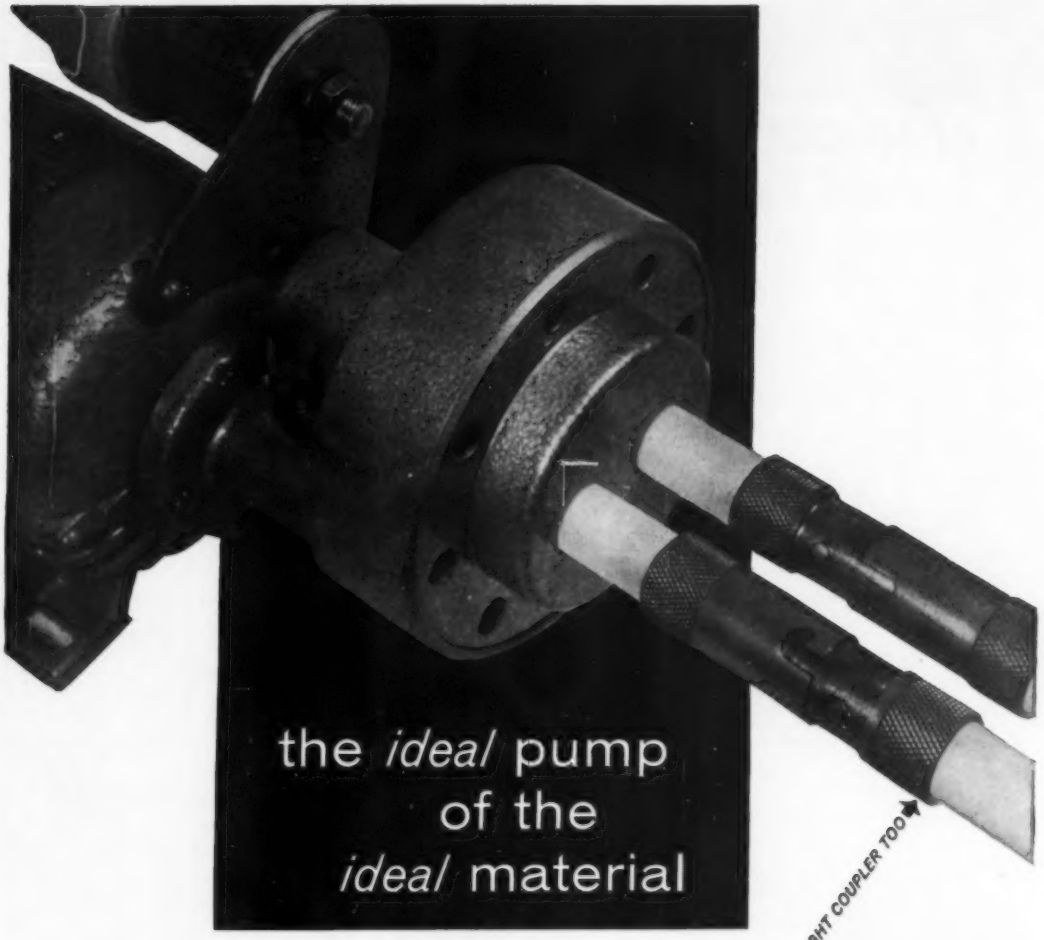

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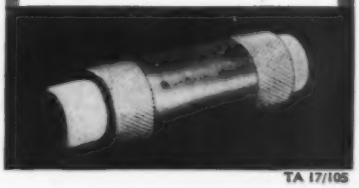

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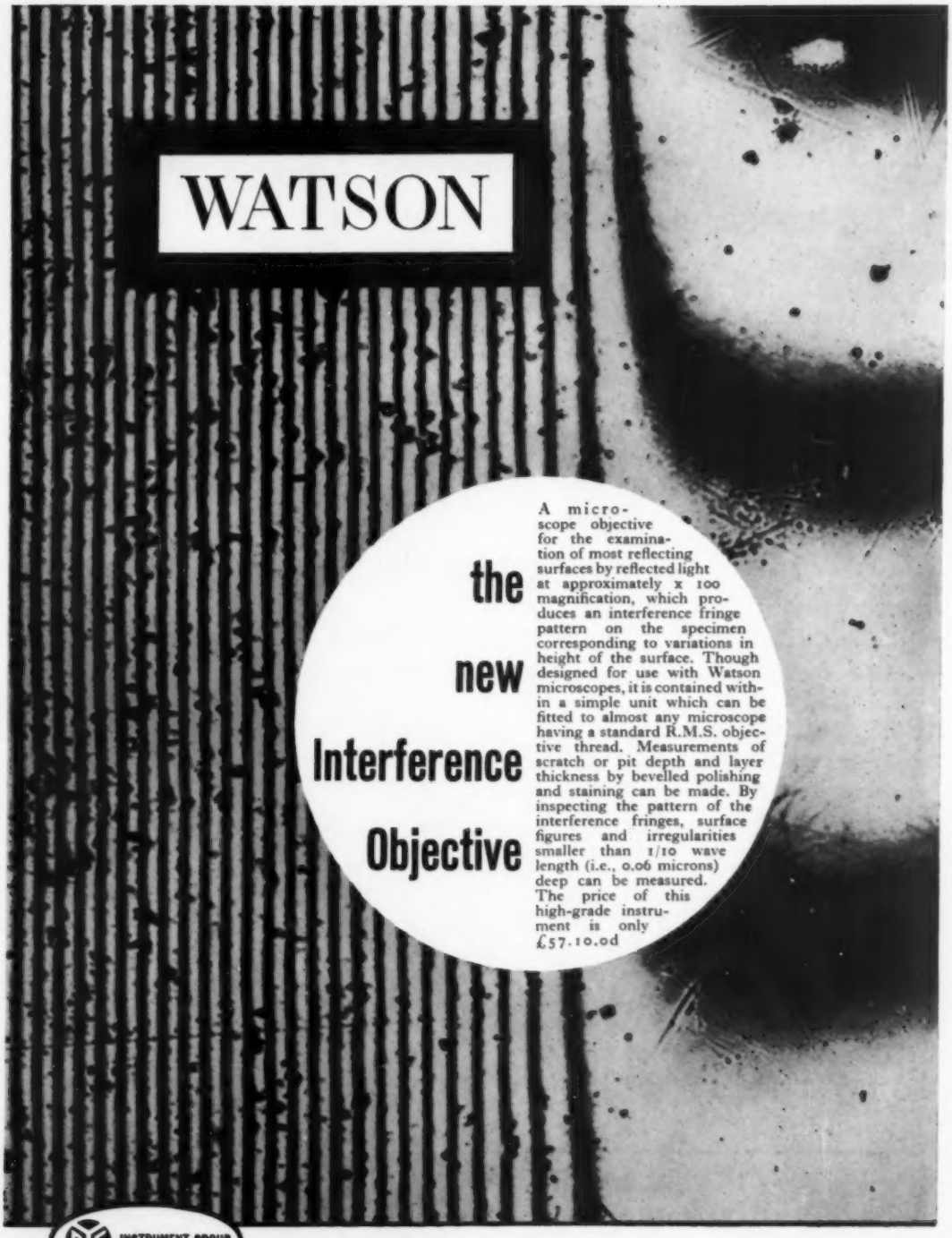
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CONTENTS

Page

EDITORIAL:

HOMO SAPIENS. 864

EXHIBITION LECTURES SERIES III—GENERAL:

THE WEISZ RING OVEN TECHNIQUE, *by W. I. Stephen* 865

SOME LABORATORY TECHNIQUES USED AT THE TIN RESEARCH INSTITUTE, *by W. E. Hoare* . 871

THE ESTABLISHMENT OF WORKSHOPS FOR LABORATORIES, *by S. F. Suffolk* 879

RHEOLOGICAL TECHNIQUES PAPER:

IMPROVED INSTRUMENT FOR THE CONTINUOUS DETERMINATION OF DYNAMIC MECHANICAL PROPERTIES OF HIGH POLYMER SOLIDS, *by P. Lord, E. R. Pithey and R. E. Wetton* 884

ARTICLE:

A SAMPLER FOR ENUMERATION OF AIR-BORNE MICRO-ORGANISMS, *by G. Grosclaude and J. Hermier* 889

Page

APPARATUS & DEVICES:

A DOUBLE-PULSE GENERATOR, *by G. Elliott* . . 893

AN APPARATUS FOR CUTTING 30-40 mm. SECTIONS OF ROOTS, *by R. G. Stickland* . . . 895

LAZY SUSAN FOR CORNER STORAGE, *by R. S. Young* 896

REPORT:

THE FIFTH INTERNATIONAL CONGRESS OF BIOCHEMISTRY, *by S. Lewin* 897

BOOK REVIEWS 902

LABORATORY EQUIPMENT AND APPLIANCES . 906

CATALOGUES, BROCHURES AND LEAFLETS . 912

MEETINGS FOR THE MONTH 912

NEWS 913

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EDITORIAL

Homo Sapiens

'WELL, I *am* surprised to see you', said His Majesty as he opened the door, letting out a fierce blast of hot air as he did so. 'I never expected to see you again after your thousand year sentence here. You have had the opportunity of enjoying all the beauties of the earth with its changing seasons, the wonders of the sky at night and the majesty of the oceans. You had an equable climate, plenty of food and drink, and all the riches of the earth. Moreover you can now benefit from all the advances of science, and I understand that the advances have been simply phenomenal in the last few years. In fact, you probably failed to recognize the old world when you got back—it must have been very different a thousand years ago. Now man is truly the lord of creation—*Homo sapiens* and all that.'

'The *Homo* part is all right,' replied Mr. Brown fanning his face, 'it is the *sapiens* part I am not so happy about. I soon came to the conclusion that all these great advances were not necessarily identical with progress, and that on balance I was better off down here'.

'Well, come and tell me all about it'. said His Majesty leaning against his favourite cauldron and sticking his pitch-fork into the molten lava, 'I must confess that your return has been a great shock to me. It upsets all my ideas for you to come back voluntarily—in fact, it's almost insulting', and he involuntarily reached out for his pitch-fork and glanced at the thermometer in the cauldron.

'Please let me explain', said Mr. Brown hastily, 'I know it sounds queer, but you will appreciate my reasons when I have explained things. Apart from the increased complexity of life and the terrific numbers of people everywhere, you don't know where you stand nowadays. In my time, the chieftains lived in the castles and stole all the best things, just as they do now, but they did at least protect you. If you were a peasant you could grow your own food in peace, and the soldiers only killed each other. It was an orderly world. There were many undesirable things but at least it made sense. You could go out, choose a bit of land, build a house, grow your crops and keep a few animals, all without let or hindrance. Today everything is licenses, taxes and officials.'

'I can sympathize with you over that', replied His Majesty, 'and things are certainly simpler here. There is only one boss, and everyone knows where he stands, even if things are painful. But have you not considered the possibility that with good conduct you might ascend to ——' he frowned and hastily corrected himself, for this had been a sore point with him ever since he had read 'Paradise Lost', and then continued '— to that other place. It may be a little colder in the celestial regions but at least it is a nice clean job polishing up the moon, planets and stars, and sending them off on their nightly journey across the sky'.

'Yes, I had thought of that,' said Mr. Brown, 'but there is one big snag. There is now an acute shortage of staff up there, and things are steadily getting more difficult. They are already working a twelve hour day, and the position is likely to get worse. Why,' he said, lowering his voice and looking round to make sure that no one was near, 'I have even heard a rumour that they are thinking of lowering the entrance standard.'

'Your mention of the moon reminds me of the last thing I heard about on earth before I left. All the big nations are going mad about sending a man to the moon. Why he should want to go, and what he could do there, are questions beyond me.'

'As a matter of fact,' observed His Majesty, 'I have been following this space research project myself, because man is beginning to poke his nose into my domain, and I am not altogether happy about it. However, the scientists say that all knowledge is valuable, and one never knows to what use any discovery may be put.'

'That may be perfectly true', acknowledged Mr. Brown, 'but this space research is quite clearly being developed for war-like purposes. If it were a peaceful or obviously a useful project the scientists would undoubtedly be starved of money. It is the intention, and the balance of things which cause me anxiety. Every few years there are devastating droughts or floods in some parts of the world, and millions starve or die of disease. Yet the leading civilized countries spend about a thousand times as much on military activities as they do on research in the conquest of disease and the production of food.'

'I have thought things over very carefully and my decision to return is no impulse of the moment. I prefer a sane and well-ordered life, albeit it a painful one, to the uncertainties of a topsy-turvy world. I know where I am here, and *Homo sapiens* is now so wise that he can blow the whole of mankind into space by pressing a few buttons, and where should I be then?'

'There are many advantages with you, Your Majesty. You provide absolute security, a steady job, central heating all the year round—and there is only one union. You are already down to a seven hour day, and I calculate you will be down to six hours in a few decades'.

'What makes you think that?' asked His Majesty.

'Why' said Mr. Brown, 'Look at the staff you've got'.

ERRATUM

In our editorial for October we stated that many of the abstracts of papers given at the Fifth International Biochemistry Congress were in Russian and so were useless to most of us. We wish to correct this statement and to say that abstracts of the Russian papers were also given in English.

EXHIBITION LECTURES SERIES III—GENERAL

THE WEISZ RING OVEN TECHNIQUE

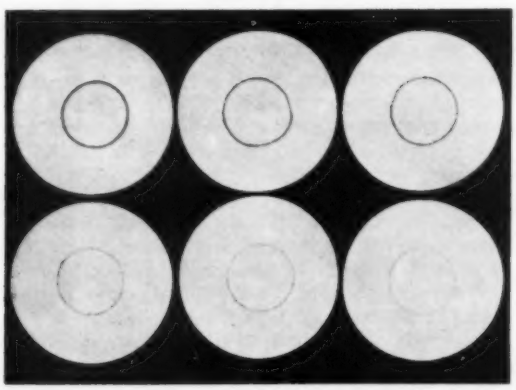
by W. I. Stephen

Lecturer in Analytical Chemistry, Birmingham University

DURING the last decade there have been few developments in the field of classical microchemical analysis which can compare with the technique known as the 'ring oven technique'. Developed by Weisz in 1954[1] as a means of qualitative inorganic analysis on sample solutions of 1 drop, the technique has been extended, principally by Weisz and his colleagues, to many other branches of chemical analysis. Although the various applications of the technique have justified the recent publication of a monograph[2], there are many who have little or no knowledge of the utility and exceptional practicability of the technique. The present author has in previous publications[3,4] detailed the principles underlying the application of the ring oven technique; despite the risk of repetition it is necessary for an understanding of the technique to repeat these briefly here.

Basically the technique is one of chemical separation of ions or groups of ions on the microgram scale. Obviously any conventional method of separation involving selective precipitation followed either by filtration or by centrifugation cannot be applied to test volumes of about 1-2 μl. However, if the process of filtration is done in a horizontal direction such as occurs when an ion is precipitated in the fibres of a filter paper, this precipitate can be washed free of other soluble ions by gradual application of a suitable wash liquid to the centre of the spot of precipitate on the paper. The capillarity of the paper ensures that a constant flow of wash liquid extends from the precipitate and covers a large area around it. If the paper is then dried the precipitated ion confined to a small area

Dr. W. I. Stephen.



Universal Standard Scale.

General view of ring oven and other apparatus.

of the paper can be removed by cutting or punching out. This is only a partial answer to the problem. The soluble ions in the filtrate are not easily recoverable; they are spread over a very large area compared with that covered by the original test drop and therefore are very considerably diluted. It may not be possible to apply tests to identify these ions because the dilution limits of the tests are exceeded. An example of this type of separation is that of iron and copper. The iron can be precipitated with ammonia, or the copper with hydrogen sulphide. In either case, one of the ions is unaffected by the precipitant and can be washed from the precipitated ion with a suitable wash liquid. But the use of too little wash liquid results in an incomplete separation while too much gives rise to the large moist

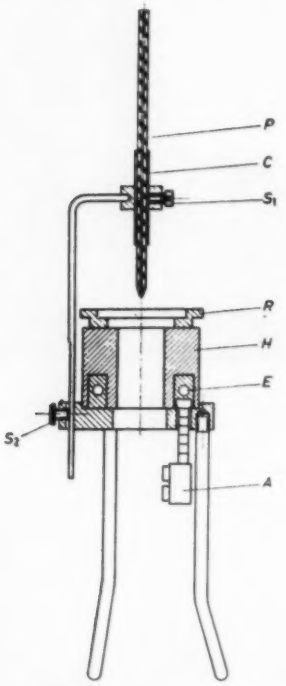


Fig. 1. Diagram of the ring oven.

area containing the soluble ion in a state of too great dilution. Only the central precipitate is in an analytically useful form for subsequent chemical identification. If, however, during the washing out process, the moist area is prevented from enlarging itself by enclosing it within a hot boundary, the solvent reaching this boundary will evaporate and all the soluble material will be retained at this boundary. This is the simple principle used by Weisz in his ring oven, shown in Fig. 1.

In its simplest form, the ring oven is a cylindrical aluminium block, H, with a central hole of 22 mm. diameter drilled out along its entire length. An annulus is cut out of the base of the block to hold a nicrome heating element, E, which is insulated from the metal with asbestos string. The block is fixed to a circular aluminium base plate supported on three legs, again with a central hole of the same diameter as in the block. The leads from the element are brought out through the base plate to a connector, A. The element has a resistance of about 30 ohms and is supplied with 24-30 volts from a variable transformer. The optimum surface temperature of the block is about 115°C. The adjustable side-arm holds a glass collar, C, which guides a capillary pipette, P, vertically above and exactly over the centre of the hole in the block. A ring of suitable ceramic or other heat-resisting material lies on the top surface of the ring oven and holds the filter paper in position during the operation. Refinements to this basic design have been described[2], a useful one being the incorporation of a small light source in a base plate on which the supporting legs of the ring oven stand. This illuminates that part of the paper covering the central hole and enables the washing-out process to be observed more carefully.

The mode of operation of the ring oven is best explained by means of the copper-iron separation already discussed. A drop of the test solution (1-2 μl.) containing little more than 0·2 μg. each of copper and iron (III) as chlorides is placed on the centre of a suitable filter paper. Experience has shown that for aqueous solutions, S. and S. Blackband 589 or Whatman No. 40, 5·5 cm. is the most satisfactory. The test drop on the paper is exposed to H_2S gas in a special apparatus (shown in Fig. 2) or treated with 1-2 drops of H_2S-acetone reagent[4]. This produces a brown fleck of copper sulphide. The paper is then placed on the ring oven and the capillary pipette, charged with 0·05 *N* HCl, is placed in its collar so that the tip of the pipette lies on the centre of the copper sulphide precipitate. The

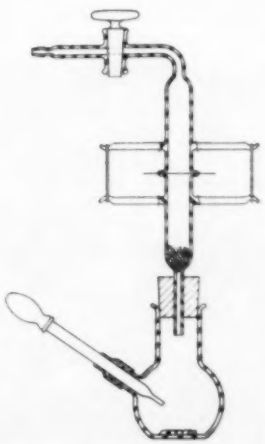


Fig. 2. Apparatus for precipitations with gaseous reagents.

paper absorbs the liquid from the pipette and a moist area spreads concentrically from the point of contact, The pipette is refilled and placed again in position this procedure being repeated 5-6 times. By this time the moist area covers the entire area of the central hole, but because of the hot metal boundary it cannot become larger. The copper sulphide precipitate is now free of iron which has been quantitatively washed to the boundary of the ring oven. The paper is then removed and dried.

The amount of iron is so small that it cannot be seen without development with a suitable chemical reagent. For this purpose the central precipitate is punched out, leaving the iron in the original paper. This is immersed in a dilute solution of potassium ferrocyanide which immediately shows up the position of the iron as a fine blue ring of Prussian blue of exactly 22 mm. diameter. The washing out process on the ring oven thus results in the transference of soluble materials to a previously determined region on the paper known as the *ring zone*. The process is quantitative; there is no indication of iron within the confines of this ring zone.

It will be remembered that the initial problem was to avoid the dilution of the materials being washed out from a precipitate. An examination of the ring zone gives some interesting and important facts. It is as thin as a pencil line. Even if it were 1 mm. thick, the area covered by it, about 66 sq. mm., is less than the area covered by the original spot on the paper, about 80 sq. mm. for a spot of 10 mm. diameter. However, with the concentrations usual in this work, the ring zone is generally 0·1-0·3 mm. thick, so that a *concentration* of from four to ten times that of the original is achieved. Furthermore, it is as easy to observe the development of a coloured arc on a sector of the paper as on the whole ring so that the paper containing the ring zone can be cut up into as many as 10 pieces each exhibiting on development an arc of about 7 mm. in length. The practical importance of these facts is that tests which fail when carried out as spot tests because of the dilution limits, may succeed as ring tests, and a sub-division of the test drop is achieved into ten parts. This enables several tests to be carried out on the original test drop, and part of the ring zone can still be retained in the undeveloped form for future reference.

The separation of the copper and iron has thus been achieved. The iron has been identified in the ring zone, and it only remains to confirm the presence of copper in the little disc punched out of the filter paper. This is simply done by fuming it over bromine water (to oxidize CuS to $CuSO_4$) and treating it with a suitable reagent to detect the Cu (II) ions, such as rubeanic acid, or α-benzoin oxime. A more elegant procedure is to place the disc on the centre of a fresh filter paper on the ring oven and wash the now soluble copper ions to the ring zone, as if the little disc were nothing but a drop of copper solution placed on the underlying paper. The separation is then truly complete; all the iron is concentrated in the first ring zone and all the copper in the second. The entire process including the development of the two ring zones is completed in 2-3 minutes.

The initial applications of the ring oven technique obviously concern qualitative analysis. Thus the so-called ring tests can be used in place of conventional spot tests. The use of ring tests avoids the difficulty of exceeding the dilution limit of the spot test reagent. Such ring tests are exceedingly sensitive, as little as 0·05 μg. of most common cations giving distinctive ring zones on development with suitable reagents. The ring zones can, of course, be divided into several sectors so that the easy sub-division of a test drop is achieved.

Normally reagents which give coloured, insoluble products are used to develop the ring zones, but this is not essential. Ordinarily soluble spot test products can be localized in the ring zone in the following way[5]. The test drop is placed in the centre of the filter paper and a drop of the particular reagent is placed between the test drop and the part of the paper where the ring zone will be formed. The test drop is washed to the ring zone in the usual way; as this occurs, the drop of reagent is also washed to the ring zone. Any soluble reaction product is thus concentrated in the ring zone and shows up as a coloured arc on the ring zone. Two or three such reactions with different reagents can be carried out in one operation. The reagent can also be made the centre spot and drops of different test solutions can be spotted at various points around the periphery of the reagent spot which is then washed to the ring zone. Such procedures enable several identification tests to be made simultaneously in one ring zone. As examples, iron (III) with thiocyanate and titanium (IV) with hydrogen peroxide give remarkably stable and well-defined ring zones containing the soluble coloured products normally produced on a spot test plate or in a micro-test tube.

The possibilities of anion analysis have not yet been fully investigated although several tests for individual anions have been described. Two of these deserve special mention because of the novel way in which demasking reactions are used. Thus extremely small quantities of the fluoride ion are detected as follows[6]. A drop of reagent solution containing the blue complex of aluminium with the red dyestuff chromazurol S is placed on filter paper and dried. The test drop containing fluoride is placed on this spot and the paper is again dried. The AlF_6^{3-} complex being stronger than the chromazurol S^- aluminium complex, the net result is the release of an amount of *free* dyestuff equivalent to the amount of fluoride present. This cannot be seen in the presence of the large excess of blue complex, but it can be washed out to a ring zone with ethanol as solvent. The ring zone of free dyestuff can then be treated with aluminium to form the stronger coloured blue complex. It is, perhaps, significant that this test does not finally depend on a comparison of the bleaching effect of the fluoride ion. The more intense the colour in the ring zone, the greater the amount of

fluoride in the sample drop. The sulphate ion can be detected in much the same way with barium rhodizonate as reagent[7]. The sulphate ion releases the rhodizonate ion which is washed to the ring zone and there developed by spraying with barium solution. Both these tests are sensitive to 0·005 μg. of the appropriate ion. They also illustrate the utility of the ring oven as a device for extractions. A very extensive range of ring tests is now available for the identification of cations.

The classical schemes of qualitative inorganic analysis can be used with the ring oven techniques. These involve the principles of separation on the ring oven described earlier, although other techniques can be introduced. For a separation into two groups, as for example the analysis of a mixture of iron, copper, and nickel, the iron and nickel can be washed to the ring zone, the copper remaining in the centre as copper sulphide. If, however, lead is also present, a separation into three groups is possible because now lead and copper which remain in the centre must be resolved. Oxidation with bromine vapour converts the sulphides to sulphates in the disc of paper punched out from the original filter paper. If this is placed on a new paper and the disc washed with aqueous ammonia, only the copper is drawn into the underlying paper as the cuprammonium ion; the lead remains in the little disc as basic lead sulphate. Thus three groups are obtained consisting of two ring zones, one of iron and nickel, the other of copper, and one disc containing the lead. Suitable identification tests can be applied to the three groups. It is possible by techniques such as these to identify as many as 18 of the commoner cations in a single drop of test solution.

It is also possible to carry out further separations once substances have been washed to ring zones. As an example, both copper and iron are contained in the ring zone and potassium ferrocyanide is to be used to detect these metals. Obviously, a separation is necessary and it is done as follows: a sector of the paper is cut out and laid on half of a fresh filter paper on the ring oven, so that the apex of the sector is 3-4 mm. within the cut edge of the paper whose centre lies directly where the wash-out pipette will touch. The ring zone in the sector of the paper is now lying 3-4 mm. within the boundary of the ring oven and if a dilute solution of ammonia and ammonium chloride is used as wash liquid, the iron will be precipitated as hydroxide in the original ring zone, but the copper will be washed to a new ring zone. The sector is removed from the ring oven, held over concentrated hydrochloric acid for a moment or two, and sprayed with potassium ferrocyanide. Two distinct parallel arcs are obtained, one of blue ferric ferrocyanide, the other of brown copper ferrocyanide, and separated from each other by a distance equal to the distance between the apex of the sector (a point which was the centre of the original filter paper) and the new point of application of the pipette. This separation is illustrated in Fig. 3.

Another variation makes use of a glass or metal plate with a hole of 12-15 mm. which is mounted on the ring

Fig. 3. Separation of iron and copper initially in the same ring zone.

oven and acts as a ring oven with a smaller bore hole. This is used to wash ions from a central spot, first to the smaller inner ring zone, then after removal of the inner ring to the normal outer ring zone using a solvent which will selectively transport only certain of the ions. As an example, let us assume that the test drop contains lead, iron, and zinc. Treatment with H_2S precipitates the lead in the centre, so that the iron and zinc can be washed to the inner ring with dilute hydrochloric acid. The iron can be fixed in this inner ring as the hydroxide by washing with ammonia solution while the zinc is transported to the outer ring zone. The three metals are then easily identified in their respective groups.

Yet another method has been described for the separation of ions already collected in a ring. The apparatus and technique devised by van Nieuwenburg[8] makes it possible to wash from the circumference of a paper back to the centre, an operation which is the reverse of the normal procedure. Two concentric glass rings are cemented on to a glass plate which has a central hole of about 25 mm. diameter. The rings form a circular trough of such dimensions that about a 3 mm. rim of a normal 55 mm. filter paper dips into the trough. This arrangement is laid on an asbestos plate having a 10 mm. hole in its centre. A long glass chimney conducts heat from a microburner to this hole in the asbestos plate. An illustration of the use of this device is the further separation of a ring zone containing aluminium, iron, cobalt and nickel. This is exposed first to ammonia vapour to precipitate the iron and aluminium, and then placed on the apparatus so that the rim of the paper dips into a dilute solution of ammonium chloride in the circular trough. The liquid flows radially inwards and carries to the centre all the soluble material from the ring zone, in this example the cobalt and nickel. The hot air channelled to the centre of the paper evaporates the solvent and concentrates the soluble material within a small circle. This part can be punched out, and the soluble material washed to a new ring zone. The apparatus is shown diagrammatically in Fig. 4.

These various possibilities make the ring oven a most valuable tool for qualitative inorganic micro-analysis. A knowledge of these few simple procedures for separations on the ring oven and of the principles of classical inorganic analysis is all that is required for quite formidable analyses to be done rapidly and effectively. There are now several examples of particular

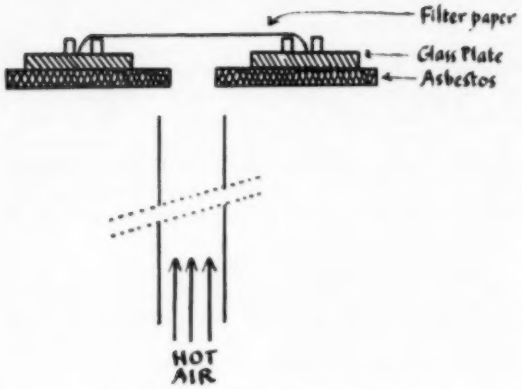


Fig. 4. Apparatus for washing ring zone back to centre.

qualitative analyses done by the ring oven techniques. Special mention must be made of the examination of bronze statues of considerable archaeological value carried out by Weisz[9], and in the field of applied analysis the ring oven has proved of value in the study of atmospheric pollution[10].

It was soon apparent that the ring oven had possibilities for quantitative as well as qualitative analysis[11]. The extremely well-defined ring zones could be compared with standard ring zones with a considerable degree of accuracy. The procedure adopted by Weisz is to prepare a 'standard scale' of a particular ion by washing 1, 2, 4, 6, 8, and 10 drops of a standard solution of the ion into ring zones. On development, these six rings form the standard scale. The procedure is repeated with exactly the same sized drops of unknown solution; three ring zones are formed from 1, 2, and 3 drops of this solution. The unknown rings are then compared visually with the standard rings and an average result is obtained for the ratio of the concentration of the unknown to that of the standard. This procedure, known as ring colorimetry, gives results which are within $\pm$ 5 per cent[12], and merely involves the preparation of individual standard scales for each ion. Thus ring zones of copper may be developed with dithio-oxamide and compared with standard rings of the copper dithio-oxamide complex, or nickel may be determined with one of the α-dioxime reagents. The possibility exists for one standard scale to serve for the analysis of a number of cations and anions[13]. This universal standard scale is prepared from an undeveloped standard scale of copper. Treatment of a copper ring zone with hydrogen sulphide results in the precipitation of copper sulphide which is neither well-defined nor stable, but if this ring zone is bathed in a solution of silver nitrate, metathesis occurs and a black, well-defined ring of silver sulphide is formed, the intensity of which is proportional to the amount of copper present. It is thus possible to determine copper quantitatively be means of these rings of silver sulphide. In the same way, other cations which give sparingly soluble sulphides can be precipitated as sulphide in the ring zone and transformed into a ring of silver sulphide. The one standard scale can then be used for comparisons with rings derived from several other cations. The result of a particular analysis is then calculated as if the ion being analysed is copper; this 'copper value' is then multiplied by a chemical factor which is simply the ratio of the equivalent weights of the unknown element and copper. The analysis of 14 metals has been described using this procedure.

Many anions form sparingly soluble silver salts which can be precipitated in the ring zone and converted to silver sulphide by treatment with hydrogen sulphide. A universal standard scale for the determination of chloride, bromide, iodide, thiocyanate, ferrocyanide, chromate and arsenate makes use of ring zones formed from a standard bromide solution *via* silver bromide and silver sulphide. Comparisons are then made in much the same way as with cations, the 'bromide value' being multiplied by an appropriate chemical factor[14].

The many possibilities for microchemical analysis presented by the ring oven technique can be further increased by combining it with other well-known methods of analysis, such as electrography, paper chromatography, polarography, autoradiography. There is not time or space to discuss these topics in detail but a brief account will serve to indicate how the development of the ring oven technique has influenced developments in established techniques.

Electrographic analysis provides a convenient means for the sampling of metallic materials. The chemical attack can be easily controlled and the effect on the specimen is hardly noticeable. The main difficulty is in the subsequent examination of the electrographic print, that is the medium into which the metal ions of the specimen are electrolysed. The use of a device as shown in the figure enables the electrolyzed metal to be retained by a little disc of filter paper which can subsequently be treated by the conventional procedures

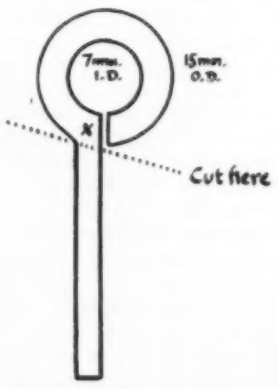


Fig. 5. Circular filter paper strip.

of qualitative analysis described in some detail above[15]. This technique of electrolytic dissolution of metals is of value in the examination of very thin metal coatings, manufactured articles, museum specimens and many other types of sample where a controlled attack is necessary to avoid damage to the material.

Only a small amount of work has been done on the combination of the ring oven technique with paper chromatography[16], although this field is extremely well suited for studies of this type. Thus, the ring oven can be of value in establishing the position of materials separated by one dimensional chromatography on a paper-strip. A novel procedure devised by Weisz is to run the chromatogram in a circular form on a piece of paper shaped as in the figure. An extremely dilute sample is applied at point A and the paper is placed on a glass hook fused to the top of a weighing bottle so that the tail of paper dips into the solvent used to develop the chromatogram. After some minutes, the solvent front will have reached the end of the circular strip. The paper is then dried and the tail portion is cut off. The remaining paper ring is placed on a fresh filter paper on the ring oven and washed with solvent. The resultant ring zone is then a projection of the various spots in the original chromatogram which have by this process now been concentrated into identifiable arcs in the ring zone. Weisz has used this procedure to separate and identify copper, cobalt and nickel in 1 drop of solution containing no more than a total of 0·07 μg. The ring zone is developed with rubeanic acid and each element shows up in the order, nickel, cobalt, copper. Such techniques have many possibilities in qualitative analysis.

The ring oven technique has also proved useful in radiochemical work. For qualitative work, no additional apparatus is necessary. The ring zones containing the active elements are simply placed on suitable X-ray film and the autoradiographs so obtained provide a permanent record of the analysis. Very small amounts of active materials can be detected in this way. An example, provided by Weisz and Scott[17], is that of a solution containing labelled SO_4^{2-}, PO_4^{3-}, or I^-. This is added to a mixture of these three ions in their inactive form which act as carriers. A drop of this solution is washed to the ring zone, and the paper dried and cut into three sectors. Each sector is then treated with a precipitant for one of the ions in question (that is, barium chloride, magnesia mixture and silver nitrate) and thoroughly washed and dried. The sectors, suitably marked to distinguish them, are placed in contact with X-ray film. Only those sectors containing ions in active forms can then give autoradiographs.

The most recent work on this aspect of the ring oven technique has been described by Weisz and Ottendorfer[18]. They have studied methods for making the autoradiographic technique at least semi-quantitative. A special apparatus has been developed which enables small sectors of the same ring zone to be exposed to X-ray film for particular intervals of time. These time intervals are arranged in a geometric progression which ensures that the blackening of the ring autoradiograph occurs in a linear fashion and results in a ring zone which shows a step-wise increase in intensity. This is called a ring chronoautoradiograph. Comparison of standard and unknown ring chronoautoradiographs enables a fairly accurate estimate of the activity of an unknown solution. This new procedure offers the possibility of measuring activities at levels of a nano-curie (10^{-9} curie) and even less with a range of error within 10 per cent.

In conclusion, it has been the purpose of this article to introduce the reader to the principles and techniques of the ring microchemistry. For further and more detailed information, the excellent monograph by Weisz[2] should be consulted.

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Fleming Fund Makes First Grant

The Trustees of the Fleming Memorial Fund for Medical Research recently announced the Fund's first grant. The University of Oxford has agreed to accept the Fund's offer of a grant of £48,000 for the Sir William Dunn School of Pathology at Oxford, headed by Sir Howard Florey, President of the Royal Society.

The grant is in connection with electron-microscopy work at the School, where the purification of penicillin was achieved and its clinical use made possible. Recently, too, cephalosporin C—a penicillin-like antibiotic active against some important penicillin-resistant bacteria—was discovered at the Sir William Dunn School of Pathology.

The Fleming Memorial Fund for Medical Research was launched in March this year in memory of Sir Alexander Fleming, discoverer of penicillin, to assist *basic* medical research. Its British and international appeal, with a target of £1 million, lasts until March 1962. So far over £370,000 has been raised. The Fund had its only London Metropolitan Area flag-day on 31 October.

The Board of Trustees of the Fund consists of: The Lord Heyworth, Chairman, Professor Sir Lindor Brown, C.B.E., F.R.S., Sir Henry D. Hancock, K.C.B., K.B.E., C.M.G., Professor Sir Hans Krebs, M.D., F.R.S., The Lord Sinclair of Cleeve, K.C.B., K.B.E.

EXHIBITION LECTURES SERIES III—GENERAL

SOME LABORATORY TECHNIQUES USED AT THE TIN RESEARCH INSTITUTE

by W. E. Hoare, D.Sc., F.I.M.

Tin Research Institute, Greenford, Middx.

The Laboratories of the Tin Research Institute exist to carry out scientific research aimed at developing new uses for tin, improving existing tin products, and improving and advising on the processes by which these are made.

The author describes some of the equipment available in the Institute's Laboratories and describes specialized techniques utilized in connection with fundamental investigations on the structure and properties of tinplate, the major outlet for the metal tin.

LAST year world production of primary tin was over 165,000 tons. The principal producing countries are Malaya, Indonesia, Bolivia, Congo, Siam, Nigeria, and China. On the other hand the principal consuming countries are the U.S.A., U.K., Germany, Japan, and France. In general, tin is not principally used in the countries where it is produced, and the metal thus forms a staple and important item of world trade. For example, in at least two producing countries tin is one of the most important export commodities and source of foreign exchange.

The principal ore of tin is cassiterite which is essentially stannic oxide. This is won by dredging alluvial deposits or by deep-rock mining and, after suitable washing and concentration, it is smelted to metallic tin by reduction with coke in large reverberatory furnaces. Following relatively simple metallurgical refining operations the metal is cast into 28 or 56 lb. ingots. A very large portion of all tin produced is of the so-called 'Standard' quality with better than 99·75 per cent purity. Very high purity brands containing over 99·90 per cent are also available at relatively small extra cost for special purposes.

Tin is a metal of considerable antiquity. It is mentioned in one or two of the early books of the Bible and lay writers indicate fair knowledge of the metal many hundreds of years before Christ. Bronze, an alloy of tin and copper, gave its name to one of the Ages of Man, and the Chinese were familiar with the valuable reflecting properties of high-tin copper alloys as early as B.C. 400. The Romans used and appreciated the quality of tin-coated food vessels, and indeed there are many interesting examples of the use our forebears made of this tractable metal.

The principal present applications of tin are alloys, chemical compounds, and protective coatings for other metals. Thus some of the important products in which the Tin Research Institute is interested are tinplate, solder, bronze, bearing alloys, electrodeposited and hot-dipped coatings of tin and tin alloys, and inorganic and organic tin chemicals.

It is of interest to consider briefly what are the basic properties of tin which have led to its widespread use, not only in ancient crafts, but also in modern technology. Tin is found in the fourth group of the periodic table. The metal is very corrosion resistant, has good appearance and reflectivity, a low melting point (232°C.) and alloys readily with many other metals. In its pure form tin is, mechanically speaking, relatively weak, but it has extremely good malleability and ductility.

The low melting point, good alloy forming characteristics, and excellent corrosion resistance are the principal properties that lead to the major application of tin, which is protective coatings for other metals. Somewhere near 45 per cent of all tin is used for coatings to protect from corrosion constructional metals like steel and copper. Tin-coated mild steel in the form of the material *tinplate*, of which some 8,000,000 tons are produced each year, is the principal example of this use.

Low melting point and good alloy forming capacity also lead to the second most important use of tin, namely as solders. These are essentially alloys of tin and lead containing 30 to 65 per cent of tin, and providing a series of joining-alloys melting at temperatures as low as 183°C.

The two most important commercial series of tin alloys are the bronzes, where tin is used as an addition to copper in amounts of 5-12 per cent to provide a series of malleable and corrosion resistant alloys of excellent toughness and hardness; and bearing alloys, essentially ternary alloys of tin, antimony, and copper, which provide in one material the diverse properties including fatigue strength, correct friction characteristics, and imbeddability which are necessary to provide a good engineering bearing material.

Although tin coatings may be very readily produced by the hot-dipping process, that is by merely dipping the metal into a bath of molten tin, they may also be successfully produced by electrodeposition, and this leads to a range of uses which includes not only pure tin coatings, but coatings of various alloys of tin, among which the most important are tin-zinc, tin-lead, and most recently tin-nickel.

The development of organotin compounds is fairly recent, and has proved to be a fascinating and rewarding field of study. The fourth group of the periodic table,

Dr. W. E. Hoare.

Dr. W. E. Hoare is a graduate of London University. He was Metallurgist with the Talbot Motor Company 1927-30, Research Engineer at the Building Research Station 1930-32, and carried out post-graduate research at Sir John Cass College, under a Fellowship sponsored by the International Tin Research and Development Council, from 1932-35. He joined the staff of the Tin Research Institute as Technical Development Officer and was later appointed Head of the Tinplate Section. He became Assistant Director in January, 1955. He was awarded the degree of Doctor of Science, D.Sc.(Eng.), in 1954.

He is co-author of the standard text-book on tinplate and is author of many research papers and books which have received very wide circulation and have been translated into many languages. He knows the world tinplate industry as few people do, and has lectured on his subject in the U.S.A., in Canada, and in most of the European countries.

He is a Fellow of the Institution of Metallurgists, an Associate of the Institution of Production Engineers, and among his Committee activities he numbers the Chairmanship of the Tinplate Committee of the British Standards Institution and of the Coatings Committee of the British Iron and Steel Research Association, and he is a Member of Council of the Institute of Sheet Metal Engineering.

in which tin is found, is headed by carbon, and the two elements naturally have certain chemical similarities. The organotin compounds are based on the tin-carbon linkage; that is, in all these compounds there is a tin atom directly linked to a carbon atom. Such compounds, of which dibutyl tin mercaptides and tributyl tin oxide are important examples, have a number of unique properties and their use in such various fields as timber preservatives, agricultural fungicides, stabilizers for plastics, and various types of catalysts is developing rapidly.

Fig. 1. Electroplating Development Laboratory. Equipment is available for all tin and tin alloy electrodeposition processes. The main switchboard is seen in the background.

Fig. 2. No. 1 Corrosion Laboratory. This laboratory houses equipment for electro-chemical corrosion studies.

Fig. 3. Part of Metallography Laboratories showing Reichert and Vickers metallographs.

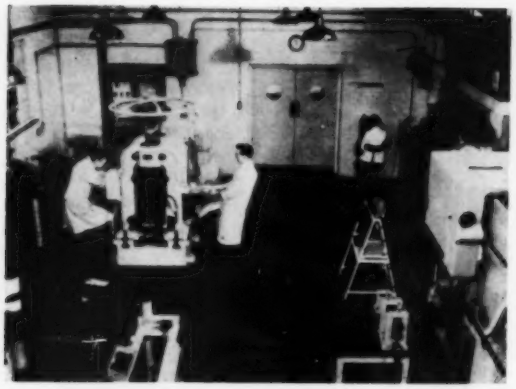

Fig. 4. Mechanical Working and Testing Laboratory showing 100 h.p. rolling mill, hard-beam tensile testing machine, hardness testers, etc.

These examples are intended to give some idea of the principal present uses of tin, but the metal in the form of its alloys or compounds has a number of other smaller but often very interesting uses ranging from the production of delicately coloured glazes for ceramics to the production of fusible alloys which may almost be held molten in the hand.

The laboratories of the Tin Research Institute exist to carry out scientific researches aimed at developing new uses for tin, improving existing tin-containing products, and improving and advising on the processes by which they are made. Our work is thus confined to a study of the *applications* of tin and we do not undertake any researches on the mining or the extractive metallurgy of the metal.

The long history of the metal means that a good deal of our effort must inevitably and rightly be concerned with the improvement and modification of processes and products: in other words, with the proper fitting of a classically useful metal into the framework of modern technology. It means, too, that absolute novelty in the field of tin applications may be difficult of access; but that it is not impossible is evidenced, for example, by recent advances in organotin chemistry and in alloy electrodeposition.

Diversity is a notable feature of tin usage and this means that the Institute must be familiar with quite a few different technologies. In fact a division of the laboratories into four departments has gradually come about. These departments are Metallurgy, Electrochemistry, Chemistry, and Electroplating. The division is not a rigorous one and the departments are distinguished principally by the training and expertise of the staff who work in them and by the equipment they utilize. It is a happy corollary of the size of the Institute that the departments are closely inter-linked, and investigations, more often than not, criss-cross between them.

On the whole our laboratories contain most of the equipment one would ordinarily expect to find in them. In Metallurgy we have mechanical working and testing equipment, metallographic and X-ray diffraction apparatus, a vacuum-casting furnace, melting and annealing furnaces, and so on. The Electrochemistry Department uses instruments and equipment for studying the fundamentals of corrosion and deposition processes, and such equipment as climatic and corrosion-test chambers. The Chemistry Department has apparatus for chemical studies including polarography, paper and gas chromatography, and absorptiometric and X-ray fluorescence analysis. The Electroplating Department is equipped with industrial-scale plating plant, the means for preparing and finishing plated articles, and for studying and controlling the electrodeposition process. We need not spend too long on items of equipment which must be deemed as reasonably familiar, but it may be of interest to see the accompanying illustrations of some of the equipment at our disposal.

Specialized Techniques

In some fields of investigation the development of special equipment or techniques has been necessary, due in some cases to the processing method under study and in others to the characteristic of the material which is required to be measured. A brief description of one or two of these techniques may be of interest to those who are concerned with laboratory practice.

Let us take as our example the material *tinplate*. Tinplate, or tin-coated mild steel sheet, is made nowadays in automatic electrodeposition lines, the complication and scale of which is such that a real laboratory-scale copy would be very costly to build and run. In order to study certain aspects of the process, and also to provide controlled experimental material for other investigations, we have therefore attempted to *simulate* certain operative characteristics of the plant rather than

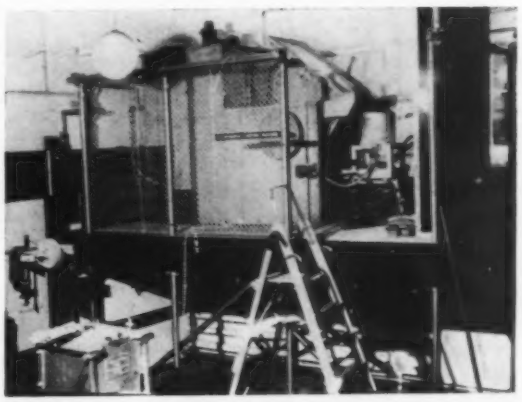

Fig. 5. Continuous casting machine for bronze.

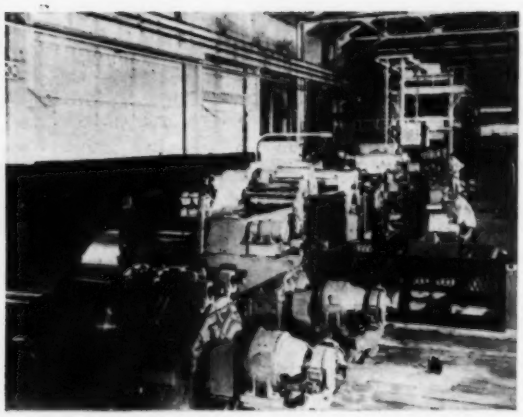

Fig. 6. Industrial plant for the continuous electro-tinning of wide steel strip. Speeds up to 1,250 ft./min. are provided for.

to make a pantographic small scale copy of the commercial plant.

'Manufacture' of Tinplate in the Laboratory

The sort of plant in which electrolytic tinplate is manufactured commercially is shown in Fig. 6. In essence, the plant comprises means for feeding wide steel strip through a series of processing units wherein it is electrolytically cleaned and etched, and then electroplated with tin. These operations are conducted at speeds between 500 and 1,500 ft. a minute, plating current densities are ordinarily in the range 300-500 A/ft.^{2} and actual plating times are normally a few seconds only.

It will be appreciated that the distinctive features of the electro-plating operation are that the cathode strip is fast-moving with respect to the electrolyte, that current densities are very high indeed, and that deposition times are so short that, for reasonable accuracy, they must be controlled to a fraction of a second. We provide these conditions by using an apparatus in which the electro-tinning is carried out in a cell *through* which the electrolyte is pumped at high speed. The adoption of a procedure wherein the electrolyte is pumped between the electrodes rather than one in which the cathode strip is driven through the plating solution results in a very considerable gain in experimental convenience. Moreover, many tests have demonstrated that both in specimen characteristics and in many aspects of electrolyte behaviour, the laboratory procedure is a good copy of industrial practice.

The apparatus illustrated in Fig. 7 consists in principle of a circuit of pipe-work connecting a reservoir, a pump, the plating cell, and a flow-meter. The reservoir is a water-jacketed neoprene rubber-lined steel tank, close to the bottom of which there is a 2 in. diameter outlet pipe leading to a pump of 80 gallons per minute capacity. In an earlier arrangement we experienced a great deal of trouble with gland packings for the pump, not only because of leakage of the rather strongly acid electrolyte but also due to organic contamination of the electrolyte, which can be rather sensitive to such things. Now we use a magnetically driven glandless pump (Hydraulic and Mechanical Developments) which avoids any risk of contamination of the electrolyte and also facilitates variation of electrolyte speed.

The plating cell is the heart of the apparatus. It consists of a box-like framework holding a milled slab of pure tin forming an anode, and a magnetic face plate on which the steel cathode is held. Anode and cathode areas are equal at 12 in. × 4 in. and the cell opening is 4·75 in. × 0·4 in., so providing a cross sectional area equal to the inside diameter of the rubber-lined 2 in. B.S.P. pipe-work. The two electrodes are clamped against gaskets into the hollow framework which is itself connected to the entry and exit pipes by suitable induction pieces. For corrosion resistance and, where necessary, for electrical insulation, all parts of the cell and pipework which come into contact with the electrolyte are either neoprene rubber coated or in solid austenitic stainless steel.

Plating current is provided by a 250 amp. 12 volt rectifier, thus permitting current densities up to 750 A/ft.^{2}. The application of the current is controlled by a process-timer and heavy-duty contactor permitting the adjustment of plating times by 0·1 sec. steps up to 10 sec. Assembly of the prepared cathode specimen into the cell is made easy by a knuckle-joint assembly, and the experimental time for a single specimen, including assembly, plating, and disassembly, is usually less than 30 sec.

The plating step does not complete the preparation of the sample because the as-plated coating must be 'flow-brightened'. This means that the tin coating must be momentarily melted and then immediately quenched. This ensures that the matte, as-deposited coating is made mirror-bright and that the coating is 'bonded' to the steel base by the formation of an alloy layer. For

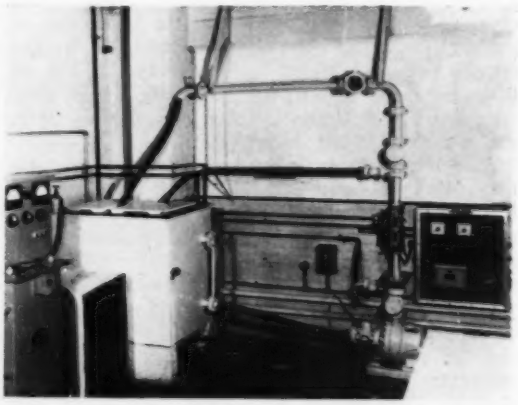

Fig. 7. Circulating electrolyte cell for investigating problems of high speed electrodeposition of tin.

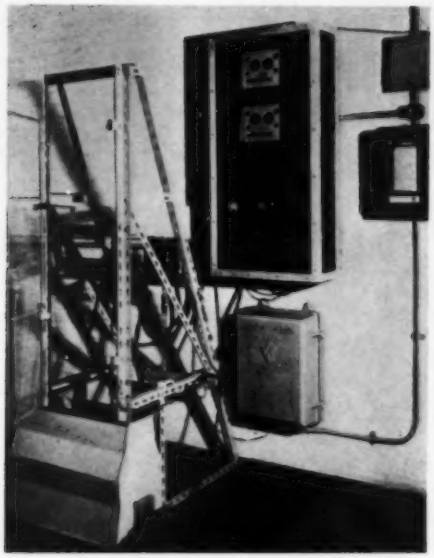

Fig. 8. Apparatus for 'flow-brightening' electro-tinned coatings. The high-speed temperature recorder is seen at right.

laboratory purposes it appeared that electrical resistance heating would be most convenient, and indeed this corresponds to the method most usually employed in commercial practice. Again the criteria are that speeds must be high, indicating short heating times and rapid quenching of the melted coating. The laboratory apparatus which has been devised for this purpose is shown in Fig. 8.

The plated specimen from the cell is clamped in a jig comprising two conductor blocks held in a suitably insulated framework. The jig slides in vertical guides so that, under its own weight, it may be allowed to fall vertically into a quench tank. Current is conveyed to the conductor blocks by heavy-duty flexible leads connected to a transformer capable of supplying, at least in short bursts, currents of the order 2,000/2,500 amp. Timing is very critical.

Basically, two non-cycling type electronic timers are employed. When the first timer is energized it closes a three-phase mains contactor operating on the primary of the transformer. This provides an initial current in the specimen of the order of 2,000 amp. At the completion of the period set on the first timer it energizes the second timer and also closes a solenoid circuit which operates a trigger releasing the specimen and allowing it to fall into the quench tank. The period set on the second timer is thus added to that set on the first timer, so permitting the timed heating period to continue during the fall of the specimen. It will be appreciated that the speed at which the specimen enters the quench can be varied very simply by altering the height of the fall, and indeed speeds closely approaching 1,000 ft./min. can be obtained. Heating power is varied by altering the voltage across the specimen by means of transformer tappings, but it will be appreciated that very careful attention has to be paid to the resistances of the various components and connections in the secondary circuit. The resistance of an average specimen is of the order of 0·001 ohms and that of the entire secondary circuit is of the order of 0·007 ohms. The smallest accidental variation of resistance in the circuit can thus greatly affect the heating current and the maximum temperature reached by the specimen.

Measurement of specimen temperature at the high heating speeds employed, which are up to 150°C. per second, obviously presents difficulties. In an earlier version maximum temperature was *detected* by an ingenious but very simple technique which is perhaps worth mentioning. This involved spot-welding 36 gauge thermocouple wires in the required position on the specimen, one wire of the couple being welded on to each face. The welding current was electronically controlled, and delicately-aligned, spring-loaded electrodes were used. The thermocouple leads were connected to a critically-damped light-spot galvanometer having a period of 0·1 sec. for full scale deflection. To record the maximum galvanometer reading a strip of photographic paper was held firmly against the scale by a light-proof backing so that the scale numbers were printed out in reverse and the maximum position of the light spot recorded.

The more sophisticated procedure now used enables a complete temperature-time curve to be plotted. The special chromel-alumel thermocouple is connected to

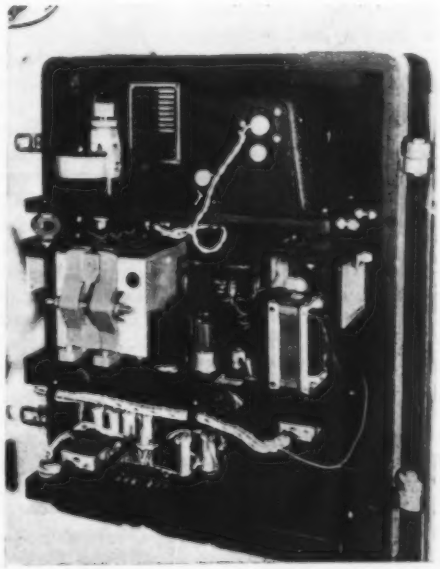

Fig. 9. Mirror galvanometer, photoelectric cell, and amplifier used for high speed temperature recording.

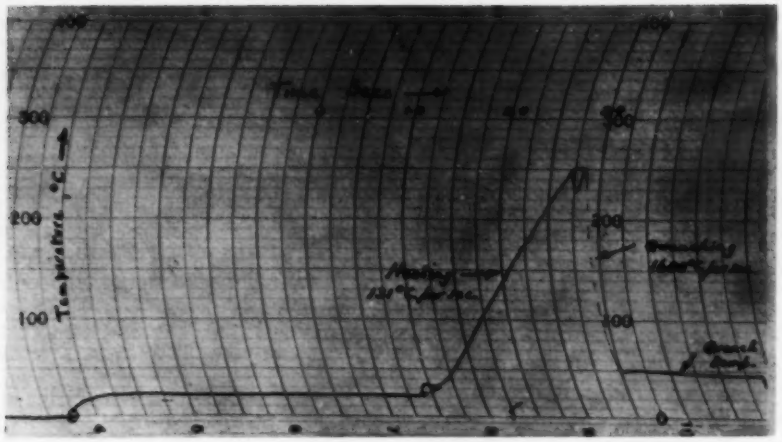

Fig. 10. Temperature record of flow-brightening cycle.

a mirror galvanometer, the reflected beam from which is directed on to a photo-electric cell. The continuous signal from this is amplified, and the output used to drive a high-speed writing device which plots the temperature time curve. The apparatus was developed by Evershed & Vignoles and the grapher is an Everett Edgcombe instrument with a response time of 0·1 sec. The general arrangement is shown in Fig. 9 and a typical temperature-time curve in Fig. 10. It will be seen that the graph gives an excellent record with a heating speed of 131°C. per sec. and a cooling rate of 1,600°C. per sec.

Appraising Tinplate Coatings

The equipment so far described is for procedures normally used to manufacture experimental tinplates in the laboratory. But the characteristics of the material are such that specialized techniques are often required to examine and appraise what has been produced. Basically the material tinplate comprises a sheet of mild steel about 0·01 in. thick, on each face of which is a coating of pure unalloyed tin approximately 50 microinches thick. Between this free tin coating and the steel base there is a layer of the iron-tin alloy $FeSn_2$ about 5 microin. thick, then there is an invisible oxide layer of thickness about 0·1 microin. and finally there is an 'oil' coating which is literally only a few molecules thick. The various layers are thus characterized by their extreme tenuity, but the overall properties of the material depend in large measure on the thickness and uniformity of the layers, and procedures for their measurement, preferably simple and rapid enough for use in routine testing, must be available.

Classical gravimetric and volumetric chemical procedures may be used for determining total tin coating thickness, and it is of interest that X-ray fluorescence is now also widely employed for this purpose. The latter method has the advantage in routine work that it is reasonably quick and non-destructive of the sample. However, when determination of both free tin and tin-iron alloy thickness is required, the methods already mentioned may be inconvenient or insufficiently accurate. The modern tendency is to use *coulometric* procedures which in fact provide a very satisfactory and elegant answer to the problem.

The technique we employ is to make a specimen of known area anodic in hydrochloric acid and to measure the quantity of electricity required to dissolve the coating layers. The method relies on the fact that the electrode potential of the specimen, measured with respect to any suitable reference electrode, changes as the coating layers are dissolved; and this potential may thus be used to indicate the exact beginning and end of the periods of dissolution of the layers of tin and tin-iron alloy,

The apparatus is shown in Fig. 11. The cell and specimen-holder are in Perspex and the cathode is a carbon rod directed at the centre of the circular specimen. The circuit comprises the test cell in series with a DC source consisting conveniently of three 12-volt batteries barretted by six 6-volt 0·25 watt bulbs, two variable resistances and a sub-standard milliammeter of range 0-50 milliamp. A high speed potentiometric recorder is connected between the specimen and a silver chloride reference electrode. Suitable characteristics of the recorder are a chart speed of 2 in. per min. and a pen speed of 4 sec. for full scale deflection. The stripping current and recorder are switched on and the current quickly adjusted to a pre-determined value which ensures anodic dissolution at 100 per cent efficiency. The potential-time curve shows two steps and the thickness of the two layers is easily determined by measuring the periods of time elapsing from the beginning of dissolution to the two changes of potential.

This equipment is essentially a set-up suitable for research purposes, as a continuous plot of the potential

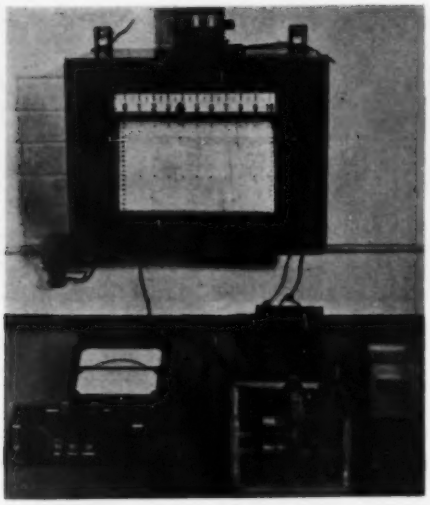

Fig. 11. Coulometric apparatus for determining thickness of tin and tin-iron alloy layers in tinplate.

change may give additional valuable information. In commercial instruments and for routine work, however, the potential changes may be used to trigger switching circuits operating a wattmeter.

The coulometric method, with some modification, is also suitable for the determination of oxide films. The principle in this case is to measure the number of coulombs required to reduce the film cathodically in an oxygen-free buffered potassium chloride solution. It is a limitation of the method that differences in composition of various oxide films are not detected and chromium oxide, which is usually present in commercially-produced passivation films, is in any case irreducible. This limitation, however, does not greatly detract from the usefulness of the technique as a control test, but clearly some discretion and experience must be used in converting the result to thickness or weight per area values. Such conversions must be empirically based and it is often better to quote the result as millicoulomb equivalents per unit area.

Determination of oil film weight involves measurements of quantities of the order of one-tenth of a gram per 1,000 sq. ft. or, say, a fraction of a microgram per sq. cm. Solvent extraction methods can be used but may involve the use of inconveniently large specimen areas, and moreover such methods give no data on local distribution. The Langmuir film spreading mechanism, however, provides a neat answer to the problem and the so-called *hydrophil balance* may be employed for determining oil-film weights in the following way.

A rectangular shallow tray is equipped with a sliding barrier resting on its edges. A light strip or dam extends almost the whole width of the tray, and has its edges attached to the tray by suitable flexible membranes. When the tray is filled with water the strip floats, and its movement, indicated by a light vane, is controlled by a torsion wire with a regulating torque head. The surface of the distilled water in the tray is swept by several movements of a slide in order to remove surface contamination and the moving slide or barrier is then placed at the end of the tray remote from the float. A sample of tinplate of known area is then dipped into the water a number of times so that the surface oil on the specimen is floated off and spreads over the water surface. The torque head is then turned through about 3° and thus deflects the indicating vane attached to the float. The barrier is now moved towards the float so compressing the oil-film, until the vane returns to the zero position. The position of the sliding barrier is an indication of the oil film weight on the sample.

A variation of this method has been developed by The Metal Box Co. and their latter procedure is regularly used in the laboratories. A specimen of area about 1,000 sq. cm., is extracted by re-fluxing in benzene and the extract is concentrated to a small known volume. A portion of the concentrate is then directed by a micrometer syringe on to a clean water surface of known area. The quantity of solution which can be applied before a permanent lens of oil appears on the water surface forms a measure of the concentration of the extract. The micrometer syringe is capable of dispensing the extract in 0·001 ml. steps.

Experimental Alloys

An example of an experimental technique used in connection with investigations on new alloys of tin may be of interest. In such studies we may be working with some of the less common and therefore expensive metals, such as titanium, and with high purity metals such as electrolytic iron, which we make ourselves and which are therefore available only in comparatively small quantities. Essentially the problem is to get as much information as possible, particularly on mechanical properties, from very little experimental material. The procedure we employ is to make our melts, usually in 50 or 100 gram quantities, in a vacuum melting and pouring furnace. The furnace, designed by the National Research and Development Corporation and manufactured in this country by Wild Barfield Ltd., is shown in Fig. 12. It permits melting, making additions to the melt, and pouring the ingot, all under high vacuum, or, more usually, under a low pressure of argon or hydrogen.

The ingots are allowed to cool in the furnace and cold-worked to about $\frac{1}{4}$ in. diameter rod in small grooved rollers. The rods are then drawn down to wire, and the finished wires, from $\frac{1}{16}$ to $\frac{1}{8}$ in. diameter, are tested in a sensitive hard-beam tensile testing machine (Fig. 13) which gives us precise values for ultimate tensile strength, yield point, and percentage elongation. Indentation hardness and grain size measurements are also carried out on the same specimens.

In systems where very reactive metals are being

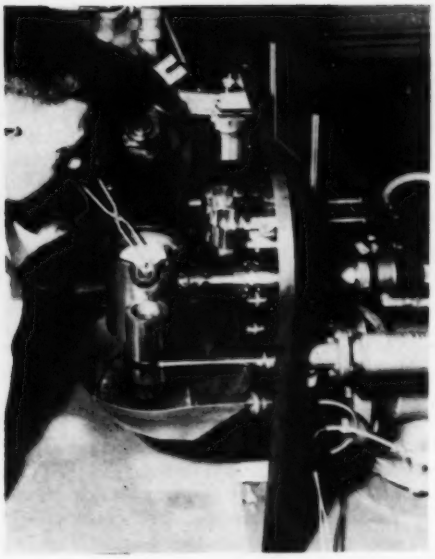

Fig. 12. Vacuum melting and pouring furnace.

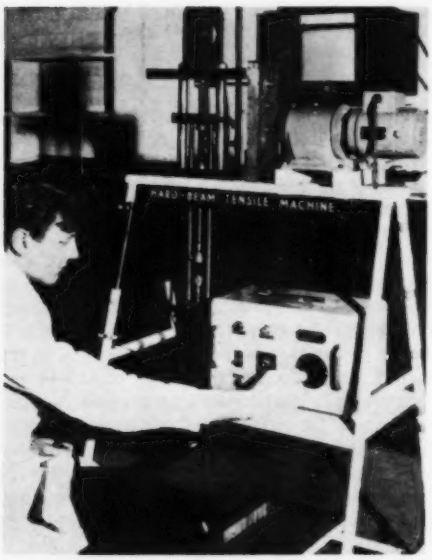

Fig. 13. Hard-beam tensile testing machine for accurate determination of stress-strain characteristics.

studied, the alloys are melted in a vacuum arc furnace on a water-cooled copper hearth, and for certain alloys the vacuum melting operation may be preceded by compacting and sintering mixed powders of the constituent metals. Subsequent working down to wire is carried out by the same procedures as are used for the vacuum cast material. We consider these techniques to be very rewarding because not only do they economize in actual experimental material, but they also enable us to by-pass the need for expensive and time-consuming machining of ordinary tensile test specimens.

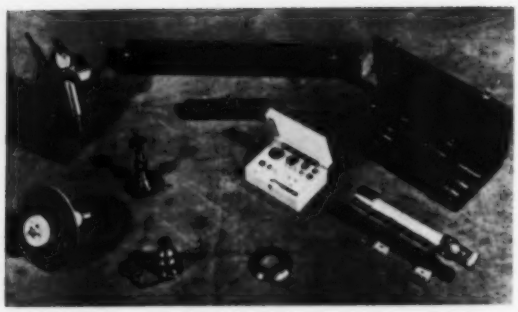

Fig. 14. Application of tin-nickel coatings in laboratory apparatus and equipment.

Tin-Nickel Alloy Coatings

Finally, mention may be made of a new material developed in our laboratories which is being increasingly used in laboratory apparatus, namely electro-deposited tin-nickel alloy. This is a plated alloy coating containing 65 per cent of tin and 35 per cent of nickel, which has a number of rather remarkable properties and is nothing at all like either of its two constituent metals. It has a pleasant appearance, is remarkably corrosion resistant, hard, stable, non-magnetic, and has good frictional characteristics. Laboratory apparatus manufacturers are making increasing use of this finish and some examples of its applications are shown in Fig. 14. It is especially suitable as a finish for analytical weights. Its success in this field is surely as good an indication as any of its stability and permanence.

AWARD OF ROYAL MEDALS

Her Majesty The Queen's gracious approval has been given to recommendations made by the Council of the Royal Society for the award of the two Royal Medals for the current year as follows:

To Sir Wilfred Le Gros Clark, F.R.S., Dr. Lee's professor of anatomy at Oxford University, in recognition of his outstanding contributions to neuroanatomy and primate morphology, which he has combined to provide new knowledge of human evolution.

To Professor C. F. Powell, F.R.S., Melville Wills professor of physics in the University of Bristol, in recognition of his pioneering work on the development of the photographic emulsion technique in the investigation of cosmic rays and the outstanding results derived therefrom on the elementary particles in cosmic radiation.

EXHIBITION LECTURES SERIES III—GENERAL

THE ESTABLISHMENT OF WORKSHOPS FOR LABORATORIES

by S. F. Suffolk, B.Sc.

Equipment Engineer, The National Institute for Research in Dairying, Shinfield, Reading, Berkshire

THERE can be few laboratories which do not possess at least the beginnings of a workshop, with a few hand tools and a soldering iron. These bare facilities often grow into something a little more ambitious when a room is set aside with a bench or two, a vice and some simple woodworking and metalworking tools with which the laboratory staff may produce the simpler pieces of apparatus, or may modify equipment which already exists.

Laboratory workshop facilities, whatever their scope, are created for one main purpose, namely to make, modify and maintain equipment for the needs of experimental work. The tools required will depend upon the type of research being carried out, some disciplines, such as physics and physiology, entailing a greater diversity of scientific equipment than others.

This paper is concerned mainly with the establishment of workshop facilities to serve a number of laboratories. It is based on experience of various laboratory workshops and particularly on the development, during the past ten years, of a centralized workshop organization at The National Institute for Research in Dairying. The objective in that case has been to design and make specialized scientific equipment needed by more than a hundred graduate staff in many fields of fundamental and applied research, and to provide a comprehensive equipment maintenance service.

Mr. S. F. Suffolk.

Organization

In a research establishment consisting of a number of laboratories scattered workshops will probably grow up, each connected with its own particular group of research workers. This is a natural development and in its early stages it may have advantages for the particular laboratories concerned. But it has distinct limitations, particularly the following:

1. The scope of any one workshop is limited by the skill of the staff of the laboratory which it serves. They are not likely to possess all the skills necessary for the maintenance of modern equipment.
2. Throughout the establishment there will be considerable duplication of workshop equipment.
3. Even if the skill is available the work of any one shop will be restricted by the range of equipment which it is possible to provide.
4. It will be necessary to call in outside help fairly frequently for the development and construction of larger projects or those of higher precision.
5. Co-ordination between workshops is difficult, restricting the free discussion of problems which arise: collaborative effort may be impeded by departmental rivalry.

When one or more of these difficulties becomes serious enough to be recognized the formation of central workshop facilities free from direct laboratory control is an obvious solution. Among the advantages of such a scheme are:

1. It will be possible to employ a wider variety of more highly skilled craftsmen.
2. The services of these skilled men will be available to all the laboratories in the establishment.
3. Unnecessary duplication of equipment will be avoided and the money saved will be available for the purchase of a wider range of equipment.
4. As the workshop staff are employed specifically for the purpose man-power and machinery will be used more efficiently.

Possible disadvantages are:

1. A less intimate contact between the laboratory and the workshop.

2. The need to establish some order of priority of work in hand.
3. The cost of having a more highly paid man in charge.

The first is not always such a disadvantage as, for instance, when the scientist has difficulty in making clear his requirements to a non-scientific technician. Much will depend on the ability of the head of the workshop to understand the ideas of his scientific colleagues and to produce practical designs in reply.

Function of the Workshop

Before any establishment sets up a central workshop, those in charge should establish quite clearly what its function is to be. Usually these functions are defined after the unit has been in existence for some time, when some difficulty has arisen and when policy may be clouded by specific problems. The primary function should be to develop new equipment which cannot be purchased but is required to meet the special needs of the laboratory. Whether design is to be included in this function will depend on the skill and experience of the research workers themselves; but this is an important facet of development for which allowance must be made.

It is to be expected that the workshop will be called upon to repair and maintain existing equipment in addition to that which it has produced. In larger organizations this second duty becomes increasingly important. If breakdowns are to be avoided it may be necessary to allocate a proportion of the workshop staff to this work to provide a system of routine servicing.

As the workshop becomes further established, giving advice on the purchase of new apparatus may become a third function and this should be encouraged. As maintenance of the equipment will fall on the engineer in charge he should be allowed to voice his preference where similar instruments are available; experience of reliability and ease of maintenance may be deciding factors.

Facilities to be provided

The call on the services of the workshop should not be underestimated. This demand appears to be unlimited but there is a physical limit to the amount of work the shops can do. To serve a real function they should be equipped with men and machines to meet any reasonable demand. The scope of services offered will vary in different establishments according to the emphasis of their research. Specialized establishments usually require specialized facilities, but more generalized groups such as the universities and research institutes need a very wide range of facilities and it is these which will be dealt with here.

The basic requirement is a machine shop and/or an instrument shop. When both are required they should be closely integrated to share tools in the middle range and to operate together on the more complex projects.

Of equal importance in these days of 'black boxes' will be electrical and electronic facilities for the development and maintenance of timers, scalers, amplifiers, and relay units which are now so common in many laboratories. It is advantageous for one of the electricians to be trained in refrigeration.

A sheet metal shop and a welding shop may be required and it is often convenient to combine the two. They should be capable of working all the common metals, including stainless steels and other corrosion-resisting alloys. At the very least, stands and boxes will be needed to support or to enclose instruments made in other shops but in many establishments the sheet metal and welding shops will also be occupied in the production of elaborate equipment.

The services of a woodworking shop are frequently advantageous. Mountings and stagings for equipment of all sizes will be required together with cabinets and cases for instruments where metal would be unsuitable or unnecessary. They can also be employed on erection of temperature-controlled rooms, ducting for ventilation schemes and special laboratory benching. Work in plastics, other than precision machining, can easily be brought within the scope of this section.

A considerable variety of stores is required by such a diverse group of tradesmen and for routine maintenance. It is essential that a properly controlled stores should be set up, with a simple form of stock control.

It should not be forgotten that services will be required to operate many of the instruments produced. Normally the provision of engineering services and the maintenance of buildings will be quite separate from laboratory workshops but it is essential that a close liaison is maintained with them, particularly when new equipment is being installed.

Layout of Workshops and Organization of work

The workshops should be laid out in such a way that there can be easy movement of work from shop to shop. For those preferring an 'open' arrangement the machine tools and sheet metal tools should be in bays opening on to a common space where the erection of the larger pieces of equipment would take place. Welding plant could be operated in this area as well as in a bay where smaller work could be carried out. A closed area would have to be set aside for precision machining and the finer instrument work, and most of the maintenance work should be carried out on one side of the main area. In addition there should be a small development laboratory. Some saving in space and cost of equipment could be effected by arranging for any services electricians and the equipment maintenance staff to share a shop and some testing equipment. Accommodation for stores should be provided adjacent to the common space; the lighter stores could be housed on a mezzanine floor over the laboratory and the instrument shop.

Small offices would be sufficient for the senior officers of the workshop and these should be as close as possible to the development laboratory. A larger room would be required for meetings to discuss

projects, provide a quiet site for writing reports and also to accommodate general reference books and files of catalogues.

Bearing in mind fire hazards and the problem of dust extraction, the woodworking section should be housed separately. In our establishment they work alongside the joiners and carpenters of the building section, with a resulting economy of machinery.

Workshop Staff

When selecting staff for workshops it is most important to choose men who are interested in producing equipment which works and not merely in taking home a pay packet at the end of the week. Good rates will have to be paid to the men required for this work, especially to those in charge of shops, as these are men who must accept responsibility as well as having skill and initiative. Industry needs such men too and can readily afford to pay highly for them.

Generally it will be necessary to recruit men who collectively will provide experience of the widest possible range of techniques. They will seldom have had experience of scientific apparatus design and time will usually be needed for them to acquire the atmosphere of experimentation with its 'one-off' jobs and continual design modifications. This emphasizes the need for a proportion of the staff to come in as apprentices or improvers.

The successful working of the group will depend on making the fullest possible use of the senior tradesmen. This will ensure that the small controlling group will have freedom to develop the more complex projects and to carry out its other functions. Minor repairs and modifications or production of simple items can be dealt with at shop level, once they have been approved. The more complex projects should be designed so that their production falls within the scope of the resources available.

Foremen must be able to bring a wide experience to bear on problems as they arise. They must also be adaptable and ready to try out and accept new techniques. Therefore they should be encouraged to widen their knowledge by attending lectures or exhibitions so that they can produce imaginative ideas and solutions.

It is most strongly recommended that foremen and senior skilled staff should be brought into the discussion of any project at a very early stage in its design. They will then understand the reason for the project and will have opportunity to suggest the best ways for carrying out the work. This co-ordinated approach will reduce the need for an expensive drawing office as most of the work can be passed to the shops in the form of good clear sketches, and the smaller items will hardly require any paper work at all.

Everything should be done to knit the group together into a team. Mutual respect and confidence at all levels are most important.

To lead this team there will be required a man with as wide experience as possible, with an ability to control labour and able to present to the team the requirements of the research staff in a form which they can readily assimilate. It will be preferable if he has had both industrial and academic experience, and if he has actually carried out research so much the better, as he will then have greater sympathy for those research workers who have difficulty in defining their requirements. He should be able to take the ideas of these scientists, representing many disciplines, and translate them into items of apparatus. Above all he should possess tact and a sense of humour; plenty of both will be required if he is to retain the respect of his colleagues.

The equipment engineer (in fact or by title) must read a wide variety of literature and be prepared to investigate new developments of technique. He should be encouraged to try out new methods even at the risk of occasional failure provided always that such experimentation is justified and purposeful.

Supervision of the Workshop

If the best use is to be made of the central workshop and its staff it will be necessary to exercise careful control of the work which is to be done. Provided that his duties have been properly defined the equipment engineer will be able to control the day to day running of the workshop. But difficulties may arise on the relative priorities to be given to larger projects competing for similar workshop facilities, and here the final decision should be governed by the relative importance of the research for which the items are needed.

Ultimate control of the workshop should therefore rest with either the head of the establishment and/or some senior committee having an overall picture of the research programme and thus able to allocate priorities. It will probably be an advantage if all projects of a certain capital value or requiring more than an agreed amount of labour for completion are referred to the committee by the equipment engineer for approval before proceeding.

Some financial reserve should be allocated to this committee so that in times of great pressure of work it may be possible to spend a little more money than was intended on special purchases. This could result in the purchasing of some standard equipment for alteration instead of the manufacture of the unit completely, or one of the more straightforward projects could be handed to an external manufacturer to produce.

Finally the committee should ensure that at all times close co-operation exists between the equipment engineer, the services engineer and the clerk of works, where such officers are employed.

The Workshop of the National Institute for Research in Dairying

This provides and maintains scientific equipment of all types for a research establishment having about 120 graduate scientists.

There is a staff of 14 skilled technicians and two apprentices under the control of the equipment

engineer who has the assistance of one graduate and one non-graduate assistant primarily concerned with development. In addition there are a stores officer, a junior assistant and one typist-clerk. Apart from advisory work and maintenance this team completes annually about 700 to 800 varied projects, most of which are small. Of the more complex items produced the following are a few recent examples to show the diversity of the work.

1. *Experimental Cheese Vat.* The Experimental Dairy required a vat of about 100 gallons capacity fitted with stirring and cutting mechanism similar to a commercial 1,000 gallon vat. Stirring rate had to be variable from 5 to 20 rev./min. and the paddles to travel 3 to 30 times per minute from end to end. The stainless steel vessel is surrounded by a water jacket for heating. Paddles and cutting knives are removable and attach to sockets on the ends of the rotating arms. (Fig. 1.)

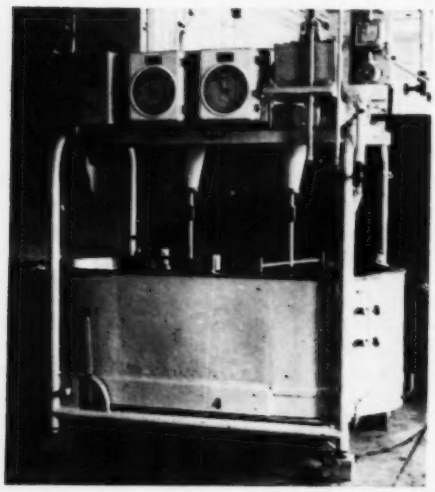

Fig. 1.

2. *Recording Viscometer.* This was required to make repeated determinations of the slowly changing viscosity of a liquid, such as renneted milk, using the U-tube method. The bulb at the top of the standard U-tube viscometer was removed and replaced with one of Perspex, with four platinum electrodes sealed in. When the liquid falls below the electrode above the bulb, a relay causes a pen to start to travel at a fixed rate across a recorder chart. When the liquid level falls below the bulb a second relay stops the pen, returns it to zero and an air pump reverses the liquid flow. The level passes the upper timing electrode to reach one at the top of the tube when the pump is switched off and the cycle repeats. Peaks of the lines drawn by the pen form a curve relating time of flow (1/viscosity) with time elapsed since the start of the experiment. (Fig. 2.)

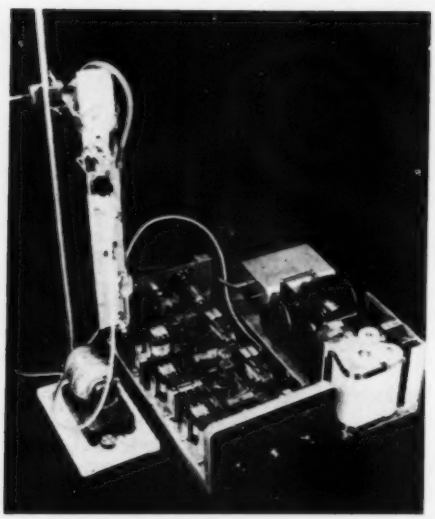

Fig. 2.

3. *Metabolism Cage.* This tubular steel structure accommodates a young pig so that the food and water intake and the liquid and solid excreta can be measured.

Fig. 3.

The problem was to devise a unit for a rapidly growing animal. The sides can be moved apart, the back moved further back and the top raised by unlocking the clamps. There is a urine collecting tray, a faeces separation tray, as well as food and water troughs which restrict spilling on to the excreta. (Fig. 3.)

4. *Claw-Piece.* One of many claw pieces which have been made for work on the milking of cows is illustrated in Fig. 4. The claw-piece hangs below the teat-cups and is the junction where the vacuum connection divides to

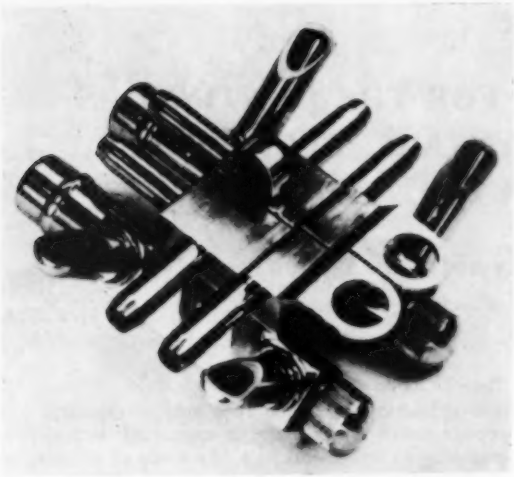

Fig. 4.

the four cups and the milk is collected and passed to the milking bucket. This example is in two similar halves, held together by a catch and allows milk from the right and left halves of the udder to be collected separately. It is made from stainless steel and all internal passages are free from bacteria-holding crevices.

5. *Shaker-Incubator*. An insulated cabinet fitted with heater, air circulating fan and temperature controller, surrounds two oscillating trays which carry baskets of test tubes. These are shaken for 24 hours as part of a method of microbiological assay of Vitamin B_{12}. The difficulty here was to obtain sufficient agitation of the tube contents from equipment which would be trouble-free over long periods with little maintenance. (Fig. 5a and 5b.)

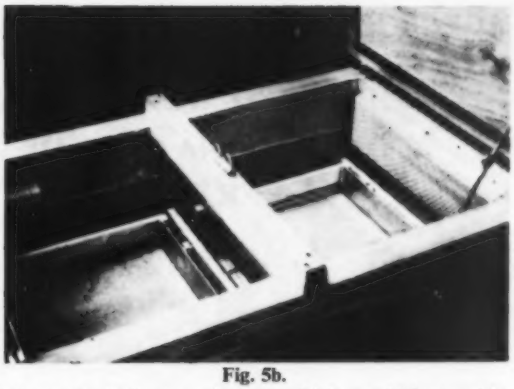

Fig. 5b.

6. *Freeze-Drier*. This is substantially similar to the freeze-drier of Record and Taylor (Biochem J. 1958, **68,** 420) except that interchangeable heads are provided. Quantities of up to 15 litres of liquid can be placed in a single stainless steel container, spun up on to the walls, frozen and dried. Smaller samples can be dried in glass or polythene bottles individually rotated in an 18-bottle head interchangeable with the 15-litre container. (Fig. 6.)

ACKNOWLEDGMENT.—*The author wishes to acknowledge the help given by Mr. H. S. Hall and Mr. R. T. Budd in preparing this paper, and to thank Mr. N. Gruber for the preparation of the photographs.*

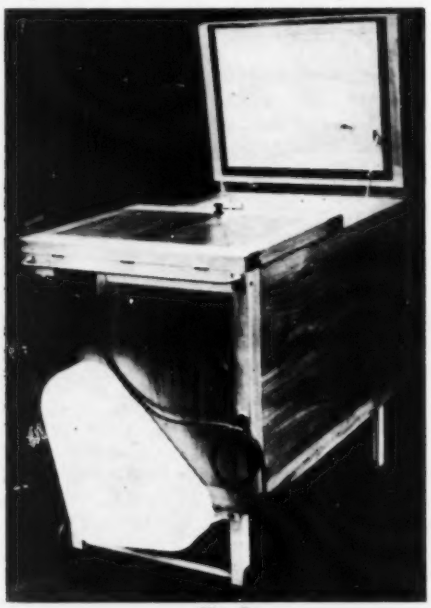

Fig. 5a.

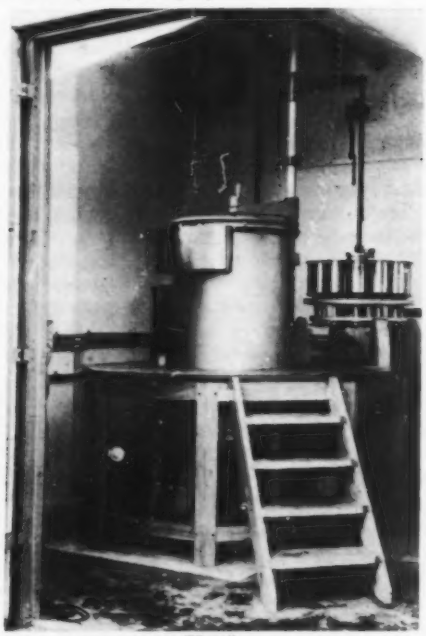

Fig. 6.

RHEOLOGICAL TECHNIQUES PAPER

IMPROVED INSTRUMENT FOR THE CONTINUOUS DETERMINATION OF DYNAMIC MECHANICAL PROPERTIES OF HIGH POLYMER SOLIDS

by P. Lord, E. R. Pithey and R. E. Wetton*

Royal College of Advanced Technology, Salford

Introduction

The prototype for this instrument was developed by Lord and Wetton. After an extensive literature survey and subsequent experimentation it was decided that any instrument designed to measure dynamic mechanical properties of polymer solids must embody the following features:

(i) Give a continuous assessment of the mechanical state of the polymer over wide ranges of temperature and frequency.
(ii) Allow reasonable accuracy on all values.
(iii) Enable all these measurements to be made on a single specimen.

With these objects in mind it was decided that an electromagnetically driven system producing torsional oscillations in a cylindrical specimen was the most suitable.

Any method of measuring the displacement of the specimen must not mechanically load the system appreciably, and, as will be shown, the moment of inertia of the driving member must be kept to a minimum. The possible methods involve either piezo-electric, photo-electric, electro-magnetic or capacitance sensitive transducers. The piezo-electric effect is temperature sensitive and therefore unsuitable, whilst the difficulty in maintaining glass surfaces moisture free, over wide ranges of temperature, rules out the photo-electric technique. Electromagnetic transducers load the system and in the presence of an electromagnetic drive are prone to spurious results. A capacitance change method, although it loads the system slightly, has been employed.

Improvements

Weaknesses not foreseeable in the original design have been eliminated where possible; the more important modifications being:

(i) Greater ease in mounting specimens.
(ii) Increased sensitivity.
(iii) Improved temperature control.
(iv) Greater stability of oscillating system.
(v) Increased accessibility.

*Now at the Dept. of Chemistry, University of Manchester.

Theory

The cylindrical specimen, 0·2 cm. in diameter and approximately 1 cm. long, is supported vertically by clamping its lower end in a pin vice and securing its upper end to a special coil situated in a magnetic field.

The equation of motion of the system in which the torque is produced by a sinusoidal current through the coil and resisted by the inertia of the system and the elasticity of the specimen is

$$I\ddot{\theta} + k\,G^*\,\theta = F_o\,e^{i\omega t} \qquad (1)$$

where I is the moment of inertia of the driving member;
θ, the angular displacement,
k, a constant dependent on the shape and dimensions of the specimen,
ω, the angular frequency,
G^* the complex modulus of elasticity,
F_o, the peak torque,
and t, the time.

The solution of equation (1) is

$$\theta = \frac{F_o\,e^{i\omega(t-\beta)}}{[(kG' - I\omega^2)^2 + k^2G''^2]^{\frac{1}{2}}} \qquad (2)$$

where

$$\tan\beta = \left[\frac{k\,G''}{k\,G' - I\omega^2}\right], \qquad (3)$$

G' is the storage modulus and G'' the loss modulus.

Of these terms β, k, I, ω, θ and F are measurable and G' and G'' can be calculated.

Considering equations (2) and (3) it is apparent that determination of the dynamic modulus over a frequency range can be divided into three frequency regions.

At low frequencies, such that the $I\omega^2$ term is small compared with $kG,'$ equation (2) reduces to

$$\theta_o = \frac{F_o}{[(kG')^2 + (kG'')^2]^{\frac{1}{2}}} \qquad (4)$$

$$\theta_o = \frac{F_o}{kG^*} \tag{5}$$

where, θ_o is the peak angular displacement. Thus from a knowledge of the peak torque and the peak displacement the complex modulus is obtained directly, k being

a shape factor of value $\frac{\pi a^4}{2l}$ for a cylindrical specimen,

with a the radius and l the length.

Also for the condition $kG' \gg Iw^2$,

$$\tan \beta = \frac{G''}{G'} = \tan \delta \tag{6}$$

the loss tangent. These quantities completely specify the dynamic properties of the polymer for a particular temperature and frequency.

At frequencies above about $\frac{1}{8}$ the resonant frequency $I\omega^2$ tends to the same order as kG' and exact solutions of (2) and (3) yield;

$$G' = 1/k\left[\frac{F_o}{\theta_o}\cos\beta + I\omega^2\right] \tag{7}$$

$$G'' = 1/k\left[\frac{F_o}{\theta_o}\sin\beta\right] \tag{8}$$

and the moduli are again given in terms of measurable quantities.

If the frequency is increased the system will pass through an amplitude resonance (in the region of 2 kc/s. for this instrument). The usual method of determining the loss tangent from the half-power points of the resonance curve is applicable in this case. Also from equation (2) $kG' = I\omega_0^2$

$$\therefore G' = \frac{I\omega_0^2}{k} \tag{9}$$

and G′ is found quite simply.

Determination of tan δ from the half-power points becomes inaccurate if tan $\delta > 0{\cdot}2$ in which case the method is as follows.

Measurement is made of θ_o and F_o when $\beta = \pi/2$, and from equation (2)

$$G' = \frac{I\omega_0^2}{k} \tag{9'}$$

$$\text{and } G'' = \frac{F_o}{k\theta_o} \tag{10}$$

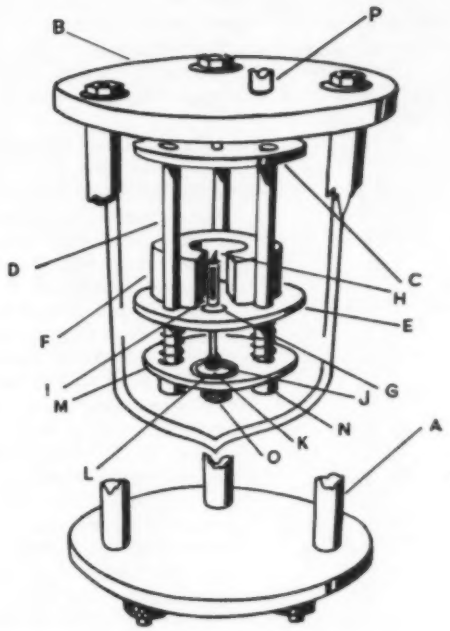

Fig. 1.

The Instrument (See Fig. 1)

The mechanical support for the loss measuring apparatus consists of three steel rods (A) (Fig. 1) 2 in. in diameter and 2 ft. long disposed at the three corners of a triangle in a steel plate $\frac{1}{2}$ in. thick and 18 in. in diameter. At the upper end of the steel rods and bolted to them is a fibre reinforced polyester plate (B) $\frac{1}{2}$ in. thick. The steel base plate carries two levelling screws and a spirit level. A slab of P.T.F.E. (C) $\frac{1}{2}$ in. thick and $3\frac{1}{2}$ in. in diameter is suspended from the polyester plate by three 6 B.A. brass bolts, the nuts of which lie recessed in holes in the top of the plate and covered with a cold setting Araldite. This construction eliminates any contact between the atmosphere and large diameter metal components, extending into the test head, and thereby reduces heat losses.

Three steel rods (D) $\frac{3}{8}$ in. in diameter and 12 in. long are suspended in a symmetrical peripheral arrangement from the P.T.F.E. slab. A brass plate (E) $3\frac{1}{2}$ in. in diameter and $\frac{3}{8}$ in. thick is held 3 in. down the rods by set screws. A magnet (F) approximately horseshoe shape is clamped to the plate with its pole pieces about $\frac{1}{2}$ in. from the edge. The capacitance transducer (G) is then arranged to lie with its axis of rotation exactly midway between the pole pieces.

Two spring loaded pivots (H) are also mounted on the same brass plate. The pivots are of hardened steel and support the coil which provides the drive to the specimen. They are set in phosphor-bronze arms $1\frac{1}{2}$ in. long and $\frac{1}{16}$ in. in diameter, which in turn are supported

by a central St. Denier rod bolted to the brass plate at the centre of the magnet. The pivots are 1 in. apart and lie midway between the pole pieces, so that when the coil is in position, it is vertical in a uniform magnetic field and able to turn through an angle of about one quarter of a radian.

Fig. 2.

The coil former (I) (exploded view in Fig. 2) is 1 in. long H shaped in cross section, cut from a circular aluminium rod $\frac{3}{16}$ in. in diameter. Into the groove are wound 150 turns of 44 gauge enamelled copper wire; this process, due to the unusual shape of the coil, is performed by hand. The lower end of the coil former fits into a ring carrying three radial vanes $\frac{3}{16}$ in. long and $\frac{3}{16}$ in. deep set at 120° to each other. The coil former is cemented to the vane holder but electrical contact between the two is maintained since the vanes form the earthed side of the capacitance transducer. The coil maintains electrical contact with the aluminium former by baring the first few turns of the coil and the former is earthed through the lower pivot which provides a convenient earth return for the input signal to the coil. The live end of the coil is in electrical contact with the pivot which carries a terminal connected to the input source.

The live side of the capacitance transducer (G) consists of three vanes set in a broad Araldite ring, inner diameter 1 in., outer diameter $1\frac{1}{2}$ in. The vanes in the Araldite ring are set in such a way as to accommodate any radial position of the earthed vanes by slight rotation and so form three parallel plate condensers.

The sensitivity of the transducer is inversely proportional to the separation between the plates which is of the order of 2×10^{-1} mm. It is desirable that, once set up, any relative motion of the plates be part of the experiment. It is for this reason that the coil is supported on spring loaded pivots, so that any slight deformation which the specimen might undergo with changes in temperature will not alter the lateral position of the coil and hence the earthed vanes.

To ensure a vertical alignment of the specimen the pin-chuck (J) is designed always to rest in the same position. It is welded to a steel base plate (K) $\frac{3}{4}$ in. in diameter and $\frac{1}{8}$ in. thick with a 1:1 tapered rim and fits exactly in a matching recess in a piece of magnetic material (L) held in a brass plate (M). The brass plate is $\frac{1}{4}$ in. thick and slides vertically over the lower ends of the three steel rods suspended from the P.T.F.E. slab, guided by three sleeves (N). Motion is achieved by a knurled nut (O) and threaded rod attached to the centre of the upper brass plate. The lower plate is lightly spring loaded which renders its motion smooth. The sleeves are accurately machined to fit the guide rods and any motion other than vertical is impossible. This affords a simple means of altering the specimen length, if necessary, since the specimen can pass completely through the pin chuck.

Through the top polyester plate passes a 1 in. diameter glass tube (P) which is connected to a vacuum line. This glass tube is also used to carry all the electrical leads from the measuring circuits to the test head. These include

- two thermocouples for temperature measurement,
- three separate leads, one for each pair of plates on the transducer, and
- a lead to the coil from the oscillator and finally a lead to earth.

Temperature Control

Accurate temperature control is obtained with a modification of an adiabatic vacuum technique described by Robinson and Oakes (1954). The system involves a special Dewar vessel which can act either as a heat insulator or as a heat exchange medium between the instrument and an external temperature bath. This is achieved by puncturing two holes in the inner wall of the Dewar flask, about one third of the way up from the bottom. This flask fits over the test head and against the underside of the polyester plate. A vacuum seal is achieved by a rubber ring set in a groove in the polyester with the same diameter as the rim of the flask. Thus with the modified flask in position the only means of heat exchange between the test head and the surrounding atmosphere is *via* the air in the flask and the polyester plate from which the whole is suspended. The reason for using a non-conducting support is to minimize heat losses.

Electrical Measurements

Except for a knowledge of the dimensions of the specimen all the required measurements are electrical. The stress is proportioned to the current flowing through the coil, and the strain to the voltage output from the capacitance change meter. The phase lag of the strain behind the stress can be determined from the phase difference between the output voltages of the oscillator and the capacitance meter. (See Fig. 3).

A variable frequency oscillator supplies an attenuated voltage (1 volt max.) to the coil, via a 10 ohm resistance. The r.m.s. current flowing through the coil is measured by determining the voltage drop across the 10 ohm resistance with a valve voltmeter.

For frequencies above 25 c/s. a Solartron (CO 540) oscillator was used in conjunction with a Solartron resolved components indicator (VP. 250). At lower frequencies a Solartron decade oscillator (OS 103) was used in conjunction with the appropriate resolved components indicator (V.P. 253).

The motion of the coil is detected by a capacitance

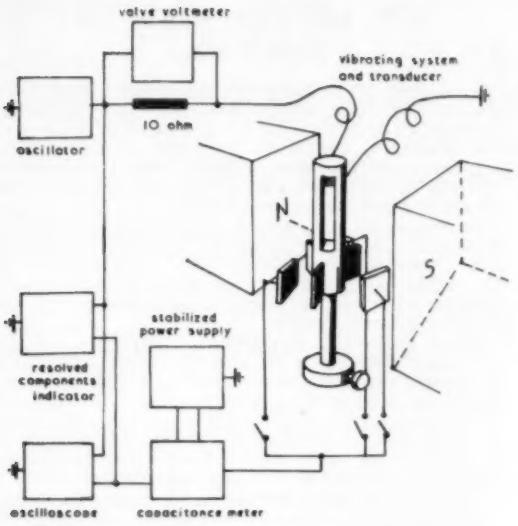

Fig. 3.

change transducer, the 'live' side of which is connected to the input of a capacitance change detection unit. (Attree, 1955). This capacitance meter produces an output voltage proportional to the change in capacitance on the input side. The output voltage is fed into the resolved components indicator, which measures the component amplitudes in phase and in quadrature with the reference oscillator voltage, i.e. the phase and amplitude of the output voltage are effectively measured. The voltages from the oscillator and the capacitance meter are monitored on a double beam oscilloscope to ensure that the signals measured were always undistorted sine waves. The presence of harmonics is not a source of error as the resolved components indicator rejects spurious frequencies.

Phase Measurements at Low Frequencies

A simple technique, requiring only the aid of a sensitive galvanometer, can be used to determine the phase lag of strain behind stress. At frequencies lower than 0·5 c/s. inductive effects in the coil are negligible and the current producing the torque in the coil is in phase with the oscillator driving voltage. The signal S, from the capacitance meter is proportional to and in phase with the strain. The first measurement is of the magnitude of S on an oscilloscope, with a known current through the coil.

The phase difference δ between the signal S and the reference voltage V can be determined by feeding S plus a standard signal C at 180° to the reference voltage to a sensitive galvanometer. This 180° signal is available on the oscillator employed and it need only be attenuated to the appropriate voltage.

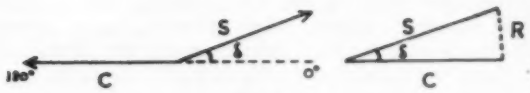

Fig. 4.

Consider the vector diagram (Fig. 4).

$$R^2 = C^2 + S^2 - 2\,C\,S\cos\delta \qquad (11)$$

$$\therefore \quad S = C\cos\delta \qquad (12)$$

is the condition under which R has its minimum value, for a set value of δ.

When this condition holds the minimum value (R_m) of the resultant R is given by

$$R_m^2 = C^2 + C^2\cos^2\delta - 2C^2\cos^2\delta \qquad (13)$$

$$= C^2(1 - \cos^2\delta)$$

$$= C^2\sin^2\delta$$

$$\therefore \quad R_m = C\sin\delta \qquad (14)$$

Thus if the signal S can be varied over the appropriate range a minimum value (R_m) can be found for the resultant voltage. Moreover this value (R_m) is directly proportional to sin δ, the required phase measurement.

R is measured as the swing of a sensitive galvanometer, earthed on one side. By varying the driving current to the coil a variation is produced in the amplitude of oscillation and hence in the output signal S, thus allowing the minimum value of R to be found. R_m is calibrated in terms of sin δ by employing signals of known phase difference.

This method gives values to within half a degree at frequencies of 0·1 c/s. and lower. At frequencies higher than this the particular 'Cambridge Spot' galvanometer used, approaches its resonant frequency, and gives false amplitude readings.

Calibration

Any measured phase difference will include a phase retardation due to the impedance of the coil and a small phase lag occurring in the capacitance meter. To calibrate the instrument carbon steel which has very low mechanical internal energy losses at normal and low temperatures is employed as a specimen. Measurement of tan δ with steel wire as specimen thus determines the phase shift occurring in the apparatus. Measurements were made over the complete frequency range and at different temperatures with some ten different wires and a satisfactory background calibration of tan δ for the instrument was thus achieved.

The calibration of the torque (stress) on the coil for particular values of the current is relatively simple. Using fine steel wire of measured rigidity modulus the angular displacement of the coil was determined by a

light-spot technique. The amplitudes for r.m.s. currents at various frequencies, well below resonance, agreed exactly with the static deflections and the torque was found to be accurately proportional to the current.

The most difficult calibration was that of voltage output on the capacitance meter for different strain amplitudes. The sensitivity of detection varies with the separation of the plates (i.e. the capacity). It can be shown that the sensitivity in volts per radian deflection should be proportional to the square of the capacitance of the transducer, i.e.

$$\frac{\delta V}{\delta \theta} = -RC^2 \qquad (15)$$

This was in fact verified by a large number of determinations. The angle was measured by a light spot technique. The capacity was measured in arbitrary units of a micrometer driven co-axial condenser, which is connected in parallel with the transducer in the capacitance change meter circuit. A value of the co-axial capacitance equivalent to the transducer could be found by switching the latter out of circuit and adjusting the co-axial condenser. The condition of equivalence was judged by a centre zero micro-ammeter, in the capacitance meter circuit which indicates correct tuning when undeflected.

The weakness of this procedure lies in the instability of the electronic circuit over the relatively long periods of time necessary for measurement.

Accuracy

It is extremely difficult to assess absolute accuracy on an instrument of this complexity. Taking manufacturers' specifications as a first guide, the resolved components indicator readings are accurate to within $\pm$ 3 per cent of full scale deflection, and voltages and currents can otherwise be determined to within $\pm$ 2 per cent. This would normally yield a limit of error in G′ given by equation (7) of approximately $\pm$ 5 per cent and a worse error in G″. However, in practice, after taking a background calibration curve for phase shift, phase measurements are reproducible to $\pm$ 0·3°. At large values of the phase angle ~ 30° this gives accuracy to $\pm$ 1 per cent in the loss tangent, and G″ is determined to $\pm$ 5 per cent. This accuracy falls as the phase angle becomes smaller. The maximum error in G′ is less dependent on the phase angle as a cos δ term is involved.

It should be emphasized that these are maximum errors. In practice results are graphically averaged and the probable errors are normally $\pm$ 3 per cent except at very low values of tan δ (~ ·05) when the error in G″ increases rapidly.

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NEW DEPUTY DIRECTOR FOR N.P.L.

The Council for Scientific and Industrial Research has appointed Dr. John Vernon Dunworth, C.B.E., as Deputy Director, National Physical Laboratory, D.S.I.R., in succession to Dr. G. Macfarlane, who is shortly to take up the appointment of Director of the Royal Radar Establishment, Malvern. Dr. Dunworth is at present Deputy Director of the Atomic Energy Establishment, Winfrith.

Dr. Dunworth, who is 44 and unmarried, was born in Manchester and educated at the Manchester Grammar School. He graduated with First Class Honours at Cambridge University in 1937 and then stayed on to do post-graduate research under the guidance of Lord Rutherford and Professor Norman Feather.

Shortly before the outbreak of war in 1939, he was invited to become a member of Sir John Cockcroft's team investigating problems in the use of radar. He continued in this work until 1944 working on 150 cm. and 10 cm. radar development mainly at the Radar Research Establishments at Christchurch and Malvern. Late in 1944 he went to Canada with Sir John Cockcroft to work with the National Research Council on atomic energy development.

Soon after the end of the war he returned to Cambridge University to take up his Fellowship at Trinity College and was appointed a Demonstrator in Physics at the Cavendish Laboratory. Later, in 1946, he rejoined Sir John Cockcroft at the Atomic Energy Research Establishment, Harwell, and since then has held a number of senior posts in the field of atomic physics and nuclear energy.

Forthcoming International Instruments, Electronics and Automation Exhibition

The 4th International Instruments, Electronics and Automation Exhibition at Olympia, London, from 28 May until 2 June, 1962 will once again be the world's greatest display from this ever expanding industry.

Covering an area of more than a quarter-of-a-million square feet, nearly 500 firms of international repute, many of them from overseas, will show instruments and electronic equipment, from mighty computers to instruments capable of weighing and measuring each one of the millions of particles contained in a 'hair's breadth', a considerable distance in modern science.

A feature of the 1962 I.E.A. will be the greater number of large stands—on an average individual stand space has increased by 20 per cent. This will ensure a finer display of the world's most up-to-date and reliable scientific instruments, and electronic equipment in first-class conditions.

Industrial Exhibitions Limited, the organizers of the 1962 Instruments, Electronics & Automation Exhibition, have already received many enquiries from intending visitors from some 20 countries overseas, and early indications are that the record-breaking overseas attendance of 4,000 visitors in 1960 will be exceeded, as well as the home attendance of more than 70,000 visitors.

The I.E.A. Exhibition 1962 will show visiting industrialists and scientists from both home and overseas, not only the latest developments in instrumentation and electronic equipment, but also the prototypes vital to industrialists and scientists in the years ahead.

ARTICLE

A SAMPLER FOR ENUMERATION OF AIR-BORNE MICRO-ORGANISMS

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Introduction

The problem of counting air-borne bacterial spores in the bottling room was encountered during a study on thermophilic spore-formers present in sterilized milk. The enumeration of air-borne spores presents a special difficulty because the vegetative cells of all bacteria present must be destroyed before an accurate spore-count can be made. In addition, spores must frequently be heat-treated to promote germination (Curran and Evans 1943). A method of collecting the spores must be devised which may be adapted to the subsequent treatments necessary, for example heating, before they are inoculated into a nutrient medium.

Three different procedures may be employed in the enumeration of air-borne micro-organisms: filtration, sedimentation or impingement. In the case of filtration, the air to be analyzed passes through a filtering medium which collects the micro-organisms; the filter is afterwards macerated in a sterile liquid from which the enumeration is made. As it is often difficult to recover the micro-organisms retained by the filter, a soluble filter of the type formulated by Pasteur (1860) may be used but the substance employed must be carefully chosen so as to avoid a subsequent inhibition of bacterial growth (Hammond 1958). In the sedimentation process (Anderson 1958; Decker *et al.* 1958) the micro-organisms either sink on to a solid culture medium, or are sprayed over it and adhere to its surface. The impingement procedure consists of injecting air into a liquid through specially constructed nozzles.

Only the filter and impingent procedures are practicable for spore enumeration because of the necessity of a pre-treatment, particularly pre-heating. The filtration process was chosen because it is easy to use and simply constructed. Our purpose was to improve Petri's technique (1888) of passing the air to be analysed through a glass tube filled with sand which may subsequently be mixed with gelatine and poured into plates. In our apparatus the air is passed through a layer of very small glass beads. The micro-organisms collect on the beads and are then suspended, by shaking, in a suitable liquid medium. If the air-sample has a high relative humidity it may be drawn through a condensation system to reduce its water content.

Although this apparatus was originally constructed for the enumeration of air-borne spores it may have a wider application. Some details of other applications are given below.

Details of the Apparatus

Fig. 1 and 2 show the principle of the air-sampler and its different parts.

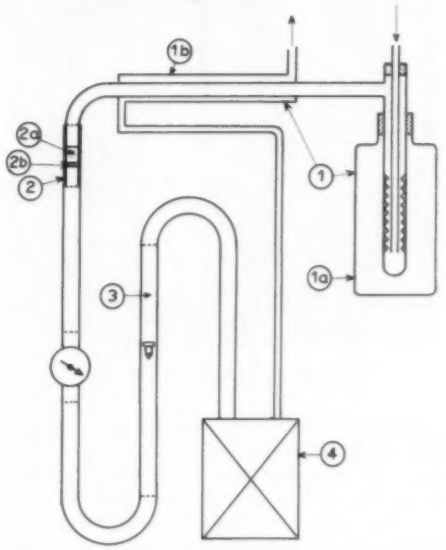

Fig. 1. Plan of the air-sampler. 1. Supplementary condensation system (1a. Cooling section; 1b. Heating section). 2. Filter (2a. Glass beads; 2b. Porous disc). 3. Flow-meter. 4. Compressor.

Filter. The filter itself consists of a brass tube 4 in. long, open at both ends, with an interior diameter of 0·6 in. A bronze porous disc*, 0·3 in. thick, with a filter threshold of at least 100 μ, is welded 4 in. from the lower end of the tube. The filter layer, 1·2 in. thick, rests on the porous disc and is composed of Ballotini No. 12 glass beads with an average diameter from 50 to 100 μ.

Air intake system. Air is drawn through the filter by a small compressor driven by an electric motor, which rests on a foam-rubber pad to prevent vibration. The air-flow from the compressor, which is between 380 to 420 cu. ft. per hour, is measured with a floating flow-meter. The compressor is connected by rubber tubes through the flow-meter to the filter (air inlet side) and to the condensation system (air exit side).

Supplementary condensation system. The condensation system includes both a cooling and a heating section. In the cooling section the air passes into a narrowed tube which leads to a brine bath at 0°C. In the heating section the dry air circulates inside a double-

*Porous disk 'Poral- classe 60', made by Ugine Carbone, Grenoble (Isère), France.

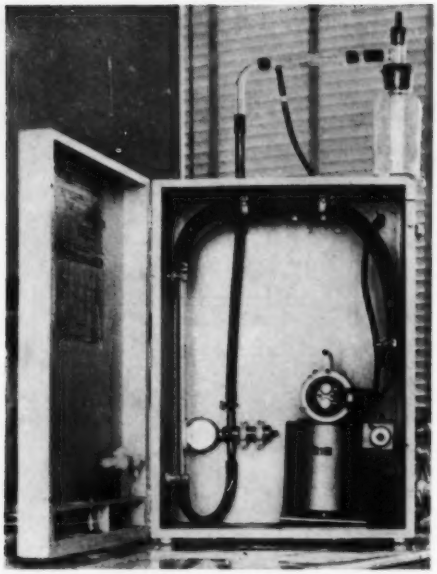

Fig. 2. Front view of the air-sampler.

walled glass tube where it is heated by air at 50°C. from the compressor circulating in the outer part of the tube.

Operation. The filter and condensation system are sterilized, the former in a dry air oven, the latter by autoclaving, and are assembled just prior to sampling. The filter and condensation system are replaced in between each sample when several samples are taken.

After sampling, the filter beads are poured into a sterile flask containing 10 ml. of a suitable diluent. The flask is then shaken vigorously by hand, or on a mechanical shaker. Any condensed water remaining in the condensation tube is poured into another sterile flask. The condensation tube is rinsed with the diluent, the final volume of the liquid being made up to 10 ml.

A plate count of the micro-organisms is made from the liquid of both flasks.

Testing the Apparatus

(1) *Operational Techniques*

Two important conditions must be fulfilled in order to obtain a quantitative count of air-borne micro-organisms. Firstly, the air passing through the filter must leave all the bacteria which it carries on the glass beads. Secondly, none of the bacteria must remain on the beads after shaking in the diluent.

To check that these two conditions were adequately met by the sampler, the air was artificially contaminated with spores of *B. subtilis*, strain SJ 2 (Plommet 1956). Air contamination was performed in the following way: 1 ml. of a spore suspension containing 10^9 spores per ml. (prepared as previously described (Hermier 1960)) was added to a half-inch layer (20-30 g.) of glass beads (Ballotini No. 12) contained in a flat-bottomed flask. The flask and its contents was then left for 24 hours in a desiccator over calcium chloride. The air was contaminated in the apparatus shown in Fig. 3. Before the

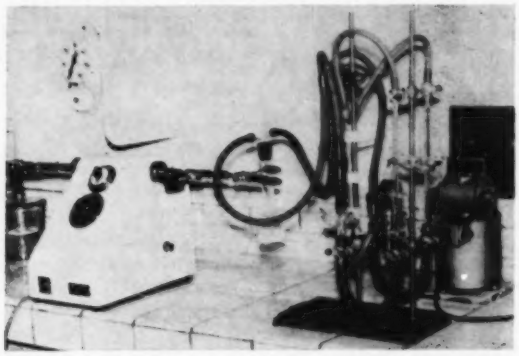

Fig. 3. Air contaminating apparatus (in this photograph, the apparatus was assembled so that the contaminated air was led through two filters mounted in parallel).

air passed into the filter it was circulated through a vertical glass tube whose pointed lower end was level with the surface of the layer of contaminated glass beads. The flask was mounted on a mechanical shaker (5 oscillations per second) with a horizontal axis. The jet of air arriving at right angles to the layer of glass beads thus throwing them to the lower part of the flask became contaminated. The stream of air which then passed through the filter contained approximately 30 spores per cu. ft.

Spores collected on the filter were shaken with glass beads in 10 ml. of 0·066 M phosphate buffer at pH 7, heated at 100°C. for 30 min. to activate germination (Hermier 1958, Plommet 1956), and inoculated into the following medium: beef extract 10 g., Evans peptone 10 g., agar 15 g., water 1,000 ml.; pH 7. The plates were incubated at 30°C. for 4 days.

(2) *Trapping ability of the filter*

The sterility of the air at the exit of the filter was tested by bubbling it into a nutrient broth which was incubated at 30°C. for 4 days, or by passing it through a second, identical, filter. Unfortunately in both cases the air-flow drops to from 3 to 5 times less than normal. Consequently a special filter, which could be completely dismantled, was used to show that all spores were trapped during normal use of the apparatus. In this filter, the porous disc was replaced by a cotton later, 0·2 in. thick, resting on a metal grid. The beads were pushed up by means of a rod, introduced into the lower part of the tube, and the successive layers were transferred into sterile beakers. The thickness of these layers was calculated from their weight. Table I gives the results of one of these tests. The results from several tests showed that the spores were always trapped in the upper ¼ in. (10 mm.) layer of glass beads.

TABLE I

The distribution of spores trapped by the filter

Layer of glass beads			*Number of spores*
N.	*Weight (mg.)*	*Height (mm.)*	
1	775	3·30	531
2	991	4·20	220
3	601	2·55	0
4	957	4·10	0
5	1,118	4·75	0
6	877	3·70	0
7	1,048	4·50	0
8	873	3·70	0
9	306	1·30	0

(3) *The release of spores into the diluent during shaking*

The following procedure was carried out to make sure that all spores collected on the filter were well suspended in the diluent during shaking.

After shaking for 10 min. in 10 ml. of phosphate buffer the liquid was poured off and the glass beads were rinsed three times in 10 ml. of buffer solution. A plate count was made of the liquid used for shaking and approximately equal quantities of the glass beads were placed in four plates and covered with nutrient agar. Since 1 ml. of the buffer solution remains on the surface of the beads after the supernatant liquid has been poured off, and assuming that all the spores are liberated into the buffer solution, the number of spores may be calculated which should be obtained in the nutrient medium inoculated with the beads. Table II shows that there is a good correlation between the plate counts and the calculated figures. Less than 1 per cent of the total number of spores remained on the surface of the beads.

TABLE II

The release of spores into the diluent during shaking

	Number of spores per 1 ml. of diluent or with glass beads	
	*Theoretical**	*Experimental*
Diluent	27·600	27·600
Glass beads	27·6	183

*See text.

Other Applications of the Apparatus

In addition to bacterial spores, this apparatus may be used for enumerating any particular type of micro-organisms or even a mixture of several species. Two examples of this are given.

Firstly it was used for testing the efficiency of an air-sterilizer using U.V. rays (Table III). The sterilizer was stopped and an aerosol of spores of *Bacillus subtilis* and *Penicillium candidum*, which are among the micro-organisms resistant to the action of U.V. rays, was sprayed into the air. A first sample of air was taken after the ventilation system was started. Then periodic air samples were taken while the sterilizer was in action. Open agar plates were placed at the side of the air-sampler for comparison. The low number of colonies found on these plates was probably due to the ventilation, which prevented the spores from settling.

The apparatus was used later to show the proportion of moulds in the air-borne microflora of a cheese-ripening room (Table IV). As the relative humidity of

TABLE III

Destruction of an aerosol of B. subtilis and P. candidum spores by U.V. air-sterilizer

Air sample			*Number of spores of*			
Hours of treatment by air-sterilizer	*Volume (cu. ft.)*	*Duration of sampling (min.)*	*B. subtilis*[1]		*P. candidum*[2]	
			per cu. ft.	*per plate*	*per cu. ft.*	*per plate*
0	15	30	167	7·5	754	1
1	27	55	1	2	211	0·5
3	29	60	0·5	2	161	0·5
17	30	60	0	0	0	0

[1]Enumeration in nutrient agar pH 7.
[2]Enumeration in nutrient agar pH 4.

TABLE IV

Study of the air in a cheese ripening room

	Number of micro-organisms per cu. ft. trapped by	
	filter	*condensation system*
Enumeration in nutrient agar at pH 4 (Moulds)	7	43
Enumeration in 'Plate Count Agar' Difco	9·5	22

the air was nearly 100 per cent the supplementary condensation system which was used on this occasion held a high proportion of the micro-organisms.

ACKNOWLEDGMENTS.—*The authors are grateful to Dr. G. Mocquot for his helpful criticism during the preparation of the manuscript and to Miss J. Altham for her valuable help in translation.*

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Recent British Standards

Laboratory deflection pH meters (B.S. 3422:1961).
A new British Standard (B.S. 3422) specifies requirements for deflection pH meters intended primarily for general laboratory use.

It covers portable and battery operated instruments, and is complementary to B.S. 3145 'Laboratory potentiometric pH meters'. It deals with both single pH scale and multi pH scale types.

As the standard is confined to the essential aspects of performance (including accuracy of calibration, stability and temperature compensation) and the interchangeability of electrodes, manufacturers are allowed wide latitude in the design of meters that will comply with the standard.

Separating and dropping funnels (B.S. 2021:1961)
When the British Standards Institution Committee revised B.S. 2021—'Separating and dropping funnels'—it came to the conclusion that the length of 'about 25 mm.' previously recommended for the portion of tube between the bulb of the funnel and the stopcock was longer than necessary and might inconvenience users carrying out certain separations.

The new edition of the standard which has just been published has therefore introduced an alternative design (Types 1B and 2B) for the spherical and conical funnels in which the bulb expands directly from the upper side of the stopcock. This is intended for use with liquids having a tendency to emulsify.

In the previous edition no special provision was made for dropping funnels with longer stems, on the grounds that they could easily be made up from the separating funnels. However, in view of the continuing demand for both categories, the spherical and conical types have now been specified as separating funnels, and the cylindrical types (plain or graduated) as dropping funnels.

None of the dimensions in this standard are considered to be so critical that they need be mandatory, and they are therefore given only as recommendations for the guidance of manufacturers in order to ensure that the funnels contain at least the nominal capacity and will withstand normal use.

Copies of these standards may be obtained from the British Standards Institution, Sales Branch, 2 Park Street, London, W.1.

AUTOMATIC CONTROL OF KNITTING QUALITY

A most important and far-reaching advance is about to take place in the art of knitting as a result of the introduction of scientific systems of 'Positive Feed' for automatic control of the knitting process. This is the prediction of Mr. W. A. Dutton, Director of Research at the Hosiery and Allied Trades Research Association, who spoke on the subject recently to a group of industrial scientists in London.

'Positive Feed' is the supply of a controlled amount of yarn to a knitting machine for each revolution or movement. Knitting variations are then virtually eliminated and this results in less wastage of manpower and materials.

Variations in length and the production of 'imperfects' have always been problems in the knitting industry. The story of how these problems are now being overcome is a striking example of how 'fundamental' as opposed to 'applied' research may, in time, assist industry to improve efficiency. When HATRA was founded in 1949, it began fundamental studies on fabric geometry. Studies of the knitted loops gradually led to the development of manual quality control instruments such as the HATRA Yarn-Speed Meter.

It was established that the best way of obtaining the same amount of yarn in each loop would be by automatic control relating the yarn feed to the machine speed. A stroboscopic method was developed which, although technically sound, proved too expensive for commercial use, as did an electronic method of control. The scientists at HATRA then investigated mechanical systems of 'Positive Feed', and by 1956 were well on the way to producing an economical and practical system, their fundamental studies of yarn and fabric geometry having given them a deeper understanding of the problem than had been available to earlier workers.

The Physical Society Exhibition Preview of Exhibits

The January, 1962 issue of *Laboratory Practice* will contain a preview of exhibits at the Physical Society Exhibition, which is to be held in the Old and New Halls of The Royal Horticultural Society, Vincent Square, London, S.W.1, January 15-19 inclusive.

A DOUBLE-PULSE GENERATOR

by G. Elliott, B.Sc., F.R.I.C.

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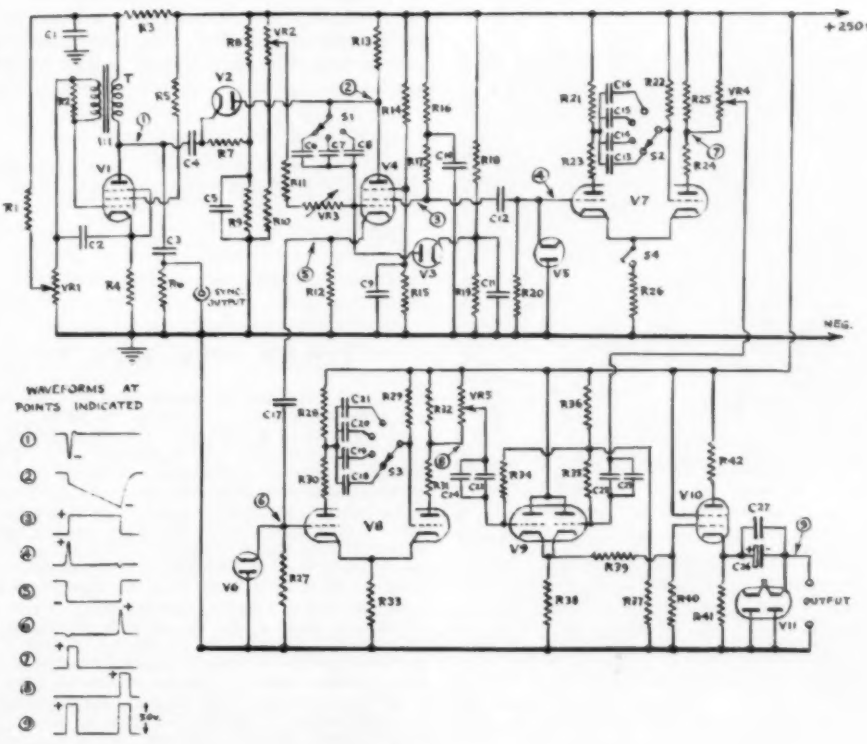
THIS note describes a versatile double-pulse generator, which has useful applications in the fields of nucleonic test equipment and semiconductor measurements. The circuit has been simplified as much as possible, and the whole unit, including the power supply (not shown in the circuit diagram), can be constructed in a small instrument case about 12 in. × 7 in. × 6 in. deep.

The unit provides single, flat-topped pulses of variable duration and amplitude, or pairs of pulses, independently adjustable for duration and amplitude and with a variable delay between pulses. A triggering output for an oscilloscope is also provided. A 6AM6 valve in a blocking oscillator, drives a phantastron delay circuit[1], using a 6F33, as well as providing external triggering pulses. The phantastron yields two triggering outputs, from the screen and the cathode respectively of the 6F33, by appropriate differentiation and clipping of the two output wave forms. The triggering pulse from the cathode is delayed with respect to that from the screen by adjusting the duration of the phantastron cycle. The undelayed pulse from the screen drives a 12AU7 in a monostable multivibrator, which generates a rectangular pulse, of a length determined by switched capacitors. The delayed pulse from the cathode of the phantastron drives an identical multivibrator to give a second pulse and the outputs from both multivibrators are combined in a 12AT7 cathode-follower mixer stage, which is used to avoid interaction between the two pulse generators. The combined signal then passes to 5763 cathode-follower output stage, with clipping diodes in the output to remove unwanted negative portions of the pulses.

Using the circuit constants shown, the repetition



Circuit diagram.

COMPONENTS LIST FOR DOUBLE PULSE GENERATOR

R1, R14, R18, 47K, 3w.
R2, 5·6K, ½w.
R3, 4·7K, 1w.
R4, R6, 220, ½w.
R5, R11, 220K, ½w.
R7, 22K, ½w.
R8, R21, R28, 15K, 1w.
R9, 33K, 2W.
R10, R36, 100K, 1w.
R12, 10K, 2w.
R13, 200K, 1w.
R15, 6·8K, 1w.
R16, R17, R19, R41, 5·6K, 1w.
R20, R25, R27 R32, R34, R35, R40, 47K, ½w.
R22, R29, 4·7M ½w.
R23, R24, R30, R31, R42, 100, ½w.
R26, R33, 2·2K. ½w.
R37, 47K, 1w.
R38, 10K. ½w.
R39. 470, ½w.
VR1, 2M. Repetition frequency
VR2, 50K. Delay time (fine)
VR3, 1M. Delay time (coarse).
VR4, VR5, 25K. Pulse amplitude.
V1, 6AM6.
V2 + V3, 6AL5.
V4, 6F33.
V5 + V6, 6AL5.
V7, V8, 12AU7.
V9, 12AT7.
V10, 5763.
V11, 6AL5.
C1, C5, C9, C10, C11, 0·1 mfd.
C2. 0·05 mfd.
C3, 100pf. mica.
C4, C14, C19. 50pf. mica.
C6, 500pf. mica.
C7, C22, C23, 1,000pf. mica.
C8, C27, 0·01 mfd. mica.
C12, C17, 300 pf. mica.
C13, C18, 20 pf. mica.
C15, C20, 200 pf. mica.
C16, C21, 1,500 pf. mica.
C24, C25, 0·25 mfd.
C26, 32 mfd. electrolytic
Working voltage of all capacitors, 300 to 500v.
S1. Delay range switch S2, S3. Pulse length switch. S4, On-off switch for 1st pulse. T 1:1 ratio transformer, Permalloy core.

frequency of the first pulse can be varied between about 20 and 5,000 c/s. The three switched ranges in the phantastron circuit give maximum delay times up to 300, 600 and 6,000 microseconds respectively. The switched ranges in the multivibrator circuits give pulse lengths of 10, 25, 50 or 500 microseconds. The output pulses are positive, with a maximum amplitude of about 50 volts and each pulse can be individually adjusted in amplitude by the potentiometer in the multivibrator circuit. The rise time of the pulses at maximum amplitude is about 0·2 microseconds, but there is some increase in the rise time as the pulses are decreased in amplitude.

The generator has a number of applications in the testing of nucleonic apparatus, for example in determining the dead time of scaling circuits and the setting up of pulse amplitude discriminators, and for these purposes the controls should be carefully calibrated, using an accurate measuring oscilloscope. The spacing between the end of the first pulse and the beginning of the second should be greater than 10 microseconds, otherwise some interaction takes place between the two waveforms. If the first multivibrator is cut out, using the switch in the cathode circuit, a single pulse output is obtained, with a variable delay from the triggering pulse. The circuit delay between the trigger output and the first pulse is sufficient to enable the leading edge of the first pulse to be observed on a fast oscilloscope.

The circuit was constructed by the author in the first instance in the laboratories of Standard Telephones and Cables Ltd., for measuring the lifetime of minority carriers in germanium single crystals by the conductivity modulation method of Spitzer *et al.*[2] For this application, the output should be taken through a series resistance of 1,000 to 2,000 ohms to provide a constant current source (the resistance of the germanium sample being less than 50 ohms in forward rectification, at all times). An E.M.I. type 3794 TA, waveform monitor was used for time and voltage measurements.

It is preferable to use a stabilized H.T. supply, to avoid fluctuations in pulse height with mains variations; this is essential for determinations of lifetime by the conductivity modulation method, where small differences in pulse height are being measured. It was found convenient to supply the whole unit from a small constant-voltage transformer, but incorporating an electronically regulated H.T. supply in the instrument case would occupy less space.

ACKNOWLEDGMENT.—*The author would like to acknowledge the help of Mr. A. H. Hooke with some of the circuit details.*

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D.S.I.R.—Does It Pay Off?

An economic and general assessment of the value of scientific and industrial research was given by Sir Harry Melville, Secretary of D.S.I.R., in a talk to the Manchester Statistical Society on November 8. He pointed to the tremendous increase of scientists and engineers working in industry and to the parallel growth of expenditure on civil research and development in the last five years. This expansion has been reflected particularly in the pattern of foreign trade—goods from the science-based industries now account for nearly two-thirds of the value of exports from this country.

On the work of D.S.I.R. itself, Sir Harry said that by its grants for research in the Universities, the Department can influence to some extent the balance of national effort in fundamental research. An increasing volume of research and development is being financed by industry within the Research Associations in which research work has grown by over 50 per cent since 1949. The total income of the Research Associations is now nearly £8 million, of which 25 per cent is contributed by D.S.I.R. Expenditure in the Department's 15 research stations, which carry out a wide variety of work in the national interest, is over £7½ million.

APPARATUS AND DEVICES

An Apparatus for Cutting 30-40 mm. Sections of Roots

by R. G. Stickland

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THE work of Heyes and Brown (1956) and of Stickland (1961) on the biochemical changes occurring along the length of pea and bean roots indicates the need to use a known length of root in any biochemical investigation. Brown and Broadbent (1950) have described a device for cutting sections of root 0·2-0·6 mm. long. Essentially this consists of a number of holes in a copper block through which the roots are pushed by rods. The advance of the rods is controlled by a screw and when the appropriate lengths of root have been exposed they are cut off with a razor. Loening (private communication) has developed a similar apparatus using several sheets of Perspex perforated with a large number of small holes through which the roots are passed. Removal of each sheet exposes a known length of root which may then be cut off with a razor as before.

However for much biochemical work pieces of root 10-20 mm. long are required, and cutting many sections of this length by the above devices would be tedious. With the apparatus described in this article 150 such sections can be cut in 10 minutes.

Figs. 1 and 2 show the cutter and its construction. In principle, a razor blade attached to a spring-loaded lever (F) descends into a narrow slit between A and B. The root to be cut is held in the path of the blade with the tip of the root just touching E, which can be adjusted from 3 to 40 mm. from the blade, thus enabling roots of different but accurately known length to be excised.

Except for the metal parts, the cutter is constructed of Perspex. The surface on which the roots are placed to be cut is composed of pieces A and B. These are attached on their under side by O and P (shown only in Fig. 2*c*) so that a slit approximately 0·5 mm. wide is left between them for the razor blade to enter. An area is removed from the top left corner of B to leave clearance for the spring, S. Pieces G and H project above A and B respectively, to which they are attached along two-fifths of their length. I and J are attached to H and serve to keep the lever, F, which holds the blade, in position. The lever is pivoted on a metal rod (4mm. diameter) which is held between G and I. Besides the hole about which it is pivoted, the lever also has two small holes about 2·5 mm. diameter through which nuts and bolts hold the razor blade, and another hole at right angles to these for securing one end of the spring, S. The space between pieces I and J allows freedom of movement for the spring. The lever is of sufficient width at the lower end for the razor blade not to project on the outer side. The lower end of the lever fits between K and M so that the blade is guided into the slit. Piece L, about 4 mm. thick, lies between K and M and serves to prevent the lever from touching B and crushing any roots. As F and L are of the same width, F is a tight fit between K and M. This can be corrected by filing away one or both sides of the end of F and at the same time minor adjustments can be made to the position of the blade in the slit. On the back of A is placed N, which serves to hold the other end of the spring. A length of metal rod ($\frac{1}{2}$ in. diameter), tapped at one end, can also be screwed to N and enables the cutter to be supported on a retort stand.

The length of root excised is determined by the position of E. This piece is fastened to C which is held against the side of D by a bolt passing through a hole in C and a slot in A. This slot, the upper edge of C and the lower edge of D (which is secured to A) are all at

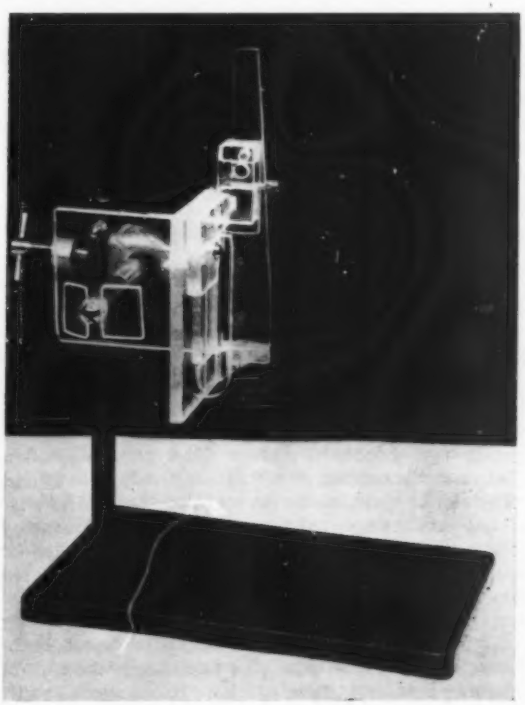

Fig. 1. View of cutter.

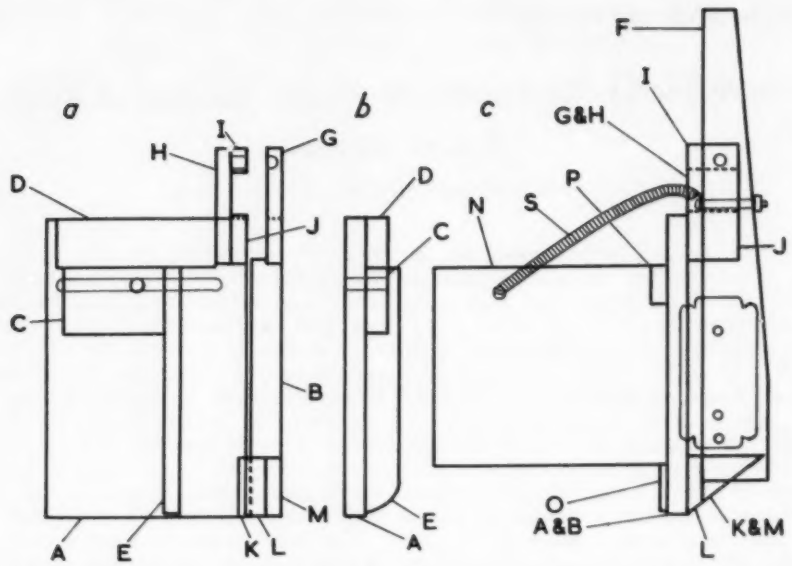

Fig. 2. Details of the cutter. (*a*) Front view. For clarity the lever (F) and its attachments, and pieces N, O and P are not shown here, but only in Fig. 2c. (*b*) side view of the left half of the cutter. (*c*) side view of the right half of the cutter. Scale x ½ approx. The lever F, is 14 cm. long.

an angle of 90° to the razor blade, so that E is parallel to the blade and can be adjusted to any position from 3 to 40 mm.from the blade.

The cutter may be used at any angle, but has been found to be most convenient with piece A vertical. When cut the roots remain attached to A by surface tension and may be pushed down with a spatula into a suitably placed beaker.

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Lazy Susan for Corner Storage

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WHEN laboratory wall benches of normal width abut in the corner of a room, a considerable amount of cupboard storage space at this location becomes unavailable, or at least inconvenient to utilize. This can be overcome by the installation of the familiar 'Lazy Susan' employed for similar storage purposes by the housewife in the kitchen. These units, with their round, rotatable, adjustable-height shelves supported on a vertical steel shaft, are obtainable from firms which sell builders' supplies.

While preferably installed during laboratory construction, they may usually be fitted under the ends of existing benches without much trouble, by temporarily removing cupboard doors or lifting a portion of the bench top.

In addition to a Lazy Susan in a corner cupboard beneath the bench, one may often be advantageously placed on top of the bench in the corner of a room. Here, by means of appropriate slots in the upper shelf, provided with some type of easy closure, long slender objects such as condensers or glass tubing may be conveniently stored vertically on what would otherwise be largely unusable bench area.

A Lazy Susan consists of several adjustable circular shelves attached to a central pivot. It can be made of either wood or metal and have either shelves or arms extending from the centre pole. The shelves or arms can be rotated in order to utilize corner-space which might otherwise be wasted.

REPORT

THE FIFTH INTERNATIONAL CONGRESS OF BIOCHEMISTRY

THREE years have elapsed since the Fourth International Congress of Biochemistry was held in Vienna. The Fifth Congress took place in Moscow from August 10 to 16. It proved very useful for recording progress in the various branches of biochemistry and particularly for the vital interchange of scientific ideas which in turn breeds new ideas. The magnitude of the undertaking of the Congress can be appreciated from the numbers involved. Over 6,000 members attended and about 3,000 papers were presented. The Congress was opened officially on the afternoon of August 10 at the Sports Stadium which was almost filled. The opening Plenary Session was addressed by Prof. D. Green on the Structure and Function of Subcellular Particles. Prof. Green gave a masterly review of the subject, and particularly of the function of mitochondria in the respiratory electron transport carried out by the study of the correlation of structural proteins, lipids and enzymes. This address was followed by a delightful concert given by the Leningrad State Symphony Orchestra.

The Congress was divided on the following day into eight Symposia and 28 Sections, all of which were held at Moscow University. The Symposia considered the topics of Biological Structure and Function at the Molecular Level, Functional Biochemistry of Cell Structures, Evolutionary Biochemistry, Molecular Basis of Enzyme Action and Inhibition, Intracellular Respiration involving Phosphorylating and Non-Phosphorylating Reactions, Mechanism of Photosynthesis, Biosynthesis of Lipids, and Biochemical Principles of the Food Industry. The 28 Sections included such widely differing subjects as the Biochemistry of Proteins and amino acids, biochemistry of antibiotics, cyto- and histo-chemistry, biochemistry of the nervous system, clinical biochemistry, biochemical genetics, biochemical pharmacology, and immunochemistry.

Because of the large number of papers, up to as many as 22 were read simultaneously. Many auditoria and lecture rooms were used, and the distances between the different assembly places were such that often it was not possible to attend certain consecutive lectures. Indeed, it is doubtful whether it was profitable to spend all one's time at the various symposia or sections, because the considerable information imparted and associated intellectual effort required inevitably resulted in a number of conscientious members developing mental indigestion. When this was superimposed on digestive tract sufferings, arising primarily from a change in diet, which affected some delegates, it was not surprising to see some in an unhappy state of mind. This state was enhanced in some individuals by the language difficulty encountered.

Language Difficulties

Language difficulties are bound to arise in International Congresses. They certainly were more acute at the present Congress than in those which have taken place in Western Countries. Outside the University very few English-speaking Russians were encountered, and lack of knowledge of the Russian language was quite a handicap. However at the Congress, simultaneous translations were available in the three official languages, English, Russian and French which could be conveniently picked up by means of transistorized short-wave receivers and heard *via* headphones, both of which were provided. In all the Sections scientific interpreters were available, and I was impressed with the speed with which the interpreters translated the questions and answers and with the understanding they displayed of the subtleties of the scientific points raised. There can be no doubt that these interpreters were carefully selected. This excellent service was needed. Prof. Cori, when announcing at the end of the Fourth International Congress in Vienna that the Fifth would take place in Moscow added that 'We have three years in which to learn Russian'. Many students of Russian present at this Congress agreed wholeheartedly, if sadly, that it takes much longer than three years to learn the language, and that considerable effort is required. Even when one has studied the Russian language at home, it is no easy matter to grasp immediately the meaning of spoken Russian. The cynic would claim that it all goes to show that higher expenditure on the teaching of Russian is not necessarily justified.

I would, however, add from personal experience that the language difficulty was not paralleled by difficulties of personal contact with Russian scientists. At the beginning of the visit it did appear that obstacles were being placed in the way of making contacts and visits to certain institutions. Some Congress members were puzzled and asked why the Russians were keen to hold the Congress in Moscow, if they were reluctant to encourage scientific contacts between Western and Russian biochemists. However, it became clear subsequently that the apparent difficulties were primarily caused by the absence on holiday of some people and by organizational difficulties arising from the larger number of Congress members than that expected. Lack of enthusiasm in making personal contacts on the part of Russian scientists below V.I.P. status may have existed at first, but once official encouragement was given, we were made welcome indeed and were shown over the institutions and the equipment we wished to see. Certainly this happened in the visit arranged to the Institute of Biochemistry where the various questions

asked were answered freely, with one exception. We were surprised to see no dogs at all in Moscow, but we heard their barking at the adjacent Pavlov Institute of Physiology. In answer to a question as to whether any dogs other than the experimental ones were to be found in Moscow, we were told that this was outside the range of biochemical questions which the Institute's spokesman was prepared to answer.

Academic Biochemical work in Russia

It is pertinent to consider academic biochemical work in Russia. The standard of biochemistry papers published in Russian journals is high, and their number is on the increase. However, I consider that the equipment I saw in the Institute of Biochemistry could be considerably augmented and improved. This Institute, headed by Prof. Oparin, is the major biochemical institution in Moscow, and has the standing of biochemical institutions in this country such as the Courtauld Institute of Biochemistry and the Chester-Beatty Institute or the Biochemistry Department at Oxford. Yet the equipment was by no means equal to that available in any of the above-mentioned British institutions. A number of ordinary Russian spectrophotometers were on view, but there were no corresponding recording instruments. Several M.S.E. and other refrigerated centrifuges could be seen, but the highest centrifugal force given was below 60,000 g. The pH meters shown were made in Hungary and their accuracy was that of 0·1 pH units; we could find no pH meter with an accuracy of 0·01 pH units or better. No recording pH meters or voltmeters or stats were seen. Only one calculating machine of Russian make, which was equivalent to the more comprehensive types of Marchant or Madas, was seen. No counter-current apparatus or the more advanced paper chromatographic apparatus was seen. I was told, however, that some more advanced equipment was available in biochemical research institutions in other places in Russia. The display of an analytical and preparatory centrifuge, similar to the corresponding one made by Beckman-Spinco, at the Exhibition (to be considered later), bears out this statement. From discussions I had with several Russian biochemists I formed the impression that the deficiencies noted were likely to be made good in the not distant future. The deficiencies, I believe, are mainly due to past lack of knowledge of the existence of such equipment in the West, rather than to the lack of money for the equipment. Increasing personal contact between Russian and Western scientists, as well as scientific exhibitions, such as the one held in conjunction with the Congress, will increase the trend to acquire such equipment, and the deficiencies could be made good within a comparatively short time. A great deal depends on the priority given. It would appear that the attainment of most things in Russia depends on priority. Once priority is given the rate of development often overtakes that of the West. This can be appreciated from consideration of Russian rocket development, the impressive building of Moscow University, the Lenin Library and the Metro of Moscow. Each of these is an outstanding achievement. In biochemical and physicobiochemical instrumentation it appears that on the whole Russia is behind the West, but there is no doubt that considerable effort is now being put into this field and, given sufficient time, the discrepancy will disappear. The contrast between the enormous advances in technology and instrumentation in the field of space research and the comparative lag in physicobiochemical instrumentation is paralleled in other fields. For example, we may contrast in this connection the outstanding development of automation in various Russian engineering and chemical industries, which depend on the production of first rate computers, with the lack of cash registers in the shops; the abacus is used everywhere. Goods are packed in paper, but tied with string; cellulose tape is unheard of.

The Exhibition

The Exhibition of Scientific Apparatus held in a building near to the University proved to be of considerable interest in that it enabled comparison of scientific equipment made in the East and the West to be made. Several Hungarian exhibits were on show. The standard of most of the exhibits was average by Western standards. An incubator, a rotary oil pump, an infra-red heater, electrically heated digesters, ordinary centrifuges and standard joint interchangeable glassware were displayed. The horizontal paper electrophoresis apparatus on show was comparatively large but allowed too much sagging of paper in the middle. The commercially introduced Western apparatus, such as that made by LKB, EEL, Shandon or Baird and Tatlock is superior. The preparative multicell ionophoretic apparatus was of a higher standard.

The Czechoslovakian apparatus on display was of greater biochemical interest and on the whole more advanced than that of the Hungarians. A line feeding device for slow continuous feeding of small volumes of liquid was exhibited. The fraction collector displayed was well designed, but not equal to the best corresponding apparatus produced in this country. The automatic amino acid analyzer was built according to the principles of Spackman, Moore and Stein.

The apparatus for batch and continuous flow culture of micro-organisms was well designed; it was evident that a great deal of attention had been paid to detail. It was composed of two main units, viz. the culture unit and the regulation and registering unit. The former was composed of three subunits which could be operated either in parallel or in series, as required. The air intake was controllable and intake of the nutrient medium was determined by a small pump. The regulation unit possessed three regulators for the supply of antifoaming agents, three temperature regulators, and

three nephelometers for continuous flow turbidity measurements (one for each of the three culture subunits), and a pH meter which could be switched automatically to measure the pH of the three cultures subunits. These and other pieces of equipment displayed were manufactured at the Instrument Development Workshops of the Czechoslovak Academy of Science.

The Tiselius apparatus displayed by Carl Zeiss Jena (East Germany) was indeed well designed and representative of the first rate optical instruments for which this firm was well known even before the Second World War. It is a compact unit to carry out determinations on electrophoretic boundary movement giving both the Schlieren and interference patterns; the experimental photographs shown were larger and clearer than those obtained with the parallel AMINCO apparatus (U.S.A.). Delivery dates quoted were within six months, if one could place reliance on the promises of many salesmen. Bitter experience makes one feel that the old joke 'there are lies, damned lies, and statistics', should be replaced by a new slogan: 'There are lies, damned lies, and equipment delivery date promises.'

Another instrument displayed by Carl Zeiss Jena was the all automatic infra-red spectrophotometer and recorder. Considerable thought and attention to detail were evident in the construction of the apparatus. Included were automatic prism changing (three prisms, KBr, NaCl and LiF) when the respective spectral regions have been traversed, and automatic scanning of regions controlled by the programme selector—set at will—thus saving considerable time in routine measurements in a series analysis.

The Japanese stand displayed a nuclear magnetic resonance spectrometer and an electron microscope, both of which were manufactured by the Japan Electron Optics Laboratory Co. Ltd. and which were well designed and equivalent to instruments made in the West.

The name Beckman-Spinco originally signified an American firm. Now however many of their products are manufactured in Germany. The apparatus displayed by the firm was almost wholly German made, but identical with that made in America. The well known preparative centrifuge (173,000 × g.) needs no introduction to biochemists. Surprisingly enough, the ultramicro analytical system displayed has not been exhibited as often as it might have been. This system, pioneered by Dr. Sanz of Geneva, combines a number of ingenious ideas and enables rapid and accurate analysis to be made in chemical and clinical laboratories. It consists of a small spectrophotometer which makes possible rapid measurements because of the fixed cuvette which is emptied automatically, at the touch of a button, into a waste-bottle. The wave-length varies from 400 to 650 millimicrons and is controlled by a wedge-interference filter having a bandwidth of 15 mμ. The microtitrator included in the set is similar in principle to an Agla microsyringe, but can be read more easily because of the large microgauge attached. The inclusion of a button-pressing system which resets the burette to zero at the start of each titration saves time and effort in that it enables the volume to be read without subtraction. The Microfuge can centrifuge 20 blood samples in 60 seconds and is switched off automatically at the end of the run. Other useful devices include the use of disposable polythene tubes, thus dispensing with washing, and reagent bottles with special attached pipettes which deliver identical quantities of fluid repeatedly.

Some British Exhibits

Several British firms exhibited their products. M.S.E. displayed their well-known refrigerated centrifuges but only a photograph of the prototype of their analytical centrifuge which is the equivalent of the Beckman-Spinco type which runs to 290,000 × g. They also displayed their two ultrasonic disintegrators, Model 60 W giving an output of 60 watts, and the larger version giving 500 watts. The larger model, besides being capable of handling larger volumes of suspensions or tissures and possessing considerably greater disintegrating capacity, possesses a titanium continuous flow assembly which is jacketted to enable accurate temperature control. Disintegrators of various makes are in general use, but evidence has been accumulated in recent years to the effect that supersonic disintegration, or the use of high speed blenders, for biological matter should be as short timed as possible because large molecules, such as DNA, tend to be adversely affected by such treatments.

Baird and Tatlock displayed a number of their instruments, such as the previously exhibited radioactive chromatographic scanner, the Analmatic proportioning pipette, and the new bench centrifuge and the universal water bath.

Joyce, Loebl & Co. exhibited their double beam automatic recording microdensitometer Mark III. The instrument is indeed well designed for densitometric measurements involved in x-ray diffraction, microspectrography and cell interferometry. The speed, accuracy and reproducibility of the instrument are quite impressive and the apparatus is a first-grade instrument. The Chromoscan recording and integrating densitometer, also manufactured by this company, is a first-grade instrument. The automatic integrator and its associated six-digit counter allow the use of higher speeds than those utilizable in pen-recording integration.

Quickfit and Quartz exhibited quite a number of their glass apparatus and standard joints. Whatman displayed a range of their papers and modified ion-exchangers. Electrothermal Engineering put on a show of their apparatus and tapes. Southern Analytical exhibited a number of their instruments including the Southern Harwell pulse polarograph, the sensitivity of detection of which was given as 10^{-8}M for reversible reductions

and 5×10^{-8}M for irreversible reductions. The apparatus and the experimental graphs presented created quite a good impression.

The Scandinavian exhibitors included LKB, Pharmacia, and Radiometer. LKB displayed their chromatographic, electrophoretic and conductiometric instruments. These are highly appreciated as good instruments and need no further comment. Radiometer of Copenhagen exhibited their polarograph, pH meter PM4 and recording pH indicator units and also their conductivity bridge which can be coupled to a 0·2 amp. recorder. Pharmacia had a colourful display of the use of Sephadex in gel filtration, and any conservative biochemists present must have been impressed with its uses.

Considering the considerable number of firms engaged in the production of electronic instrumentation in radioactive work in biochemistry, it was surprising to find that only one firm, Tracerlab (U.S.A.), exhibited. Windowless counting systems, automatic counting systems and other systems were amongst the instruments shown. These were well designed and pleasantly finished.

Paper chromatographic glass apparatus was displayed by VEB Glaswerke (Thuringia, Germany). Corresponding British manufacturers would find it worthwhile to examine their competitors' well finished and well designed apparatus. Other German apparatus displayed included that of the Serva Entwicklungs Labor (Heidelberg, Germany) which displayed beautifully coloured ion exchange systems for use in the purification of water. Here again corresponding British firms would find these attractive arrangements worthy of consideration.

The Russian Exhibits

Let us consider now that part of the Exhibition which many biochemists from the West side of the Iron Curtain were anxious to see, that is the display of Russian chemical and biochemical equipment. The recent Russian Exhibition or Fair in London displayed some scientific equipment which was clearly of good and of comparatively recent design, e.g. the nuclear apparatus corresponding to that of the Harwell Zeta. The apparatus exhibited in the Fair gave the impression of being well-used but certainly not as well finished as the latest and best Western scientific apparatus. One was therefore led to believe that only average utilitarian equipment was being exhibited, and that the best was not on display. Would much more be learnt from the Congress Exhibition?

The Russian section was comparatively small. There was a number of very ordinary exhibits. For example, ordinary simple analytical balances were displayed, but no single pan or direct measuring all-enclosed weight balances. One or two radioactive measuring instruments of the elementary student type were on show and gave the appearance of having been well used; but the more advanced instrumentation normally available in research laboratories, such as exhibited by Tracerlab, was absent. However, a vacuum refrigerated centrifuge capable of 45,000 × g. and 400 ml. total capacity was displayed, and according to the person in charge the instrument was generally available in Russia. A preparative analytical centrifuge capable of speeds giving 300,000 × g. showed that in this field Russia possessed instrumentation equivalent to that of the West. According to the demonstrator, two such machines existed and more were being produced. Another advanced instrument displayed was the preparative apparatus for continuous electrophoresis in a stabilized medium. This apparatus can separate about 100 ml. of solution at a controlled temperature range of -3°C. to $+7$°C. Other large and well used spectrometric equipment and two small recording systems were displayed along with large photographs of apparatus.

The Russian section of the Congress Exhibition was on the whole a disappointment and a puzzle. Here was an International Exhibition associated with the International Biochemistry Congress, and the Russians were obviously very keen to convene the Congress in Moscow and to impress the visiting foreign scientists. One expected to see a most impressive exhibition of Russian instrumentation in chemical and biochemical fields. However, as the officially supported and planned venture of a country with outstanding technological attainments, the Russian part of the Exhibition was unbelievably small and unexceptional. The viewer may have wondered whether this was the limit of Russian achievement in this field, or whether this was the limit of what they were willing to show. It is also possible that the Russian planning of their section of the Exhibition was simply inadequate and that they did not appreciate the wide interest that would be shown in their scientific equipment. Above all one should include in one's assessment the attitude towards priorities and the question of pushing sales which contrast the Russian and Western attitudes in the manufacture of scientific instruments. It appears that the Russians are as yet not particularly concerned with the 'finish' or polish of their instruments, and in general, place external appearances low on the scale of their priorities. The lack of finish on a number of Russian instruments could therefore be explained in terms of their utilitarian outlook and on the lack of a need for sales pushing. The small scale of their part in the Exhibition was partly due to their present lag behind the Western counterparts in chemical scientific instrument manufacture, as was also evidenced in the Institute of Biochemistry, and partly to inadequate appreciation of foreign interest. The Russians' lag in their biochemical instrumentation is merely temporary. Given priority they could easily outstrip the West within a short period of time. This may not take place before the next International Congress of Biochemistry. Thereafter we may well see a change in Russian attitude from that of preparedness to buy to that of sales drive.

Considerable Progress

Returning to the Congress itself, it can be said that considerable progress was recorded in all the fields covered. However, while increasingly faster rate of progress was reported than in any previous three year period, no major discoveries were announced. In other words, in the last three years the various biochemical formulations and interpretations underwent a considerably accelerated evolution, but no revolution.

It has been accepted for the last decade that the basic genetic carrier in all biological material is nucleic acid. This is supported by various investigations such as those concerned in the ability of chemically isolated nucleic acids to initiate viral infections and to transform the genetic properties of bacteria. In many viruses and in some bacteria it is a ribonucleic acid (RNA), while in more advanced forms of life it is a deoxyribonucleic acid (DNA). The genetic information is coded in terms of the constituent purines and pyrimidines and in their sequence in the nucleic acid in double stranded helical structures, or in the single stranded polymers. Mutations are considered as genetic changes arising from alterations in the particular purines or pyrimidines and in their sequence along the nucleic acid chain. Considerable evidence has been accumulated in the last three years in support of this interpretation. In particular the investigations of Schram, Fraenkel-Conrat and their co-workers deserve mention. They investigated the effect of the treatment of genetic TMV RNA with nitrous acid (which results in deamination which transforms the constituent cytosine into uracil) and showed that this results in a detectable mutation in the virus. It has also been established by other investigators that some of the acridine dyes which associate with the phosphate backbone of the nucleic acids, e.g. proflavine, can cause mutations. Ethylating agents such as diethyl sulphate and substances which react with nucleotides of cytosine and uracil are known to be mutagenic. The importance of these researches lies in their correlation of the mutagenic result with the location in the nucleic acid.

Man has struggled throughout his history to adapt the external surroundings including living organisms to his requirements. Success has been attained in this direction by various means, including selective breeding. The finding that it is possible to correlate the mutation of *lower* living organisms by chemical treatment with the structure of the particular genetic carrier may not appear, to some, as an outstanding development. However, the significance of chemically planned and a forced evolution is of overriding importance. For millions of years evolution has been haphazard; its 'natural selection' was probably associated with the loss of useful organisms which did not happen to possess the requisites in the struggle against their natural surroundings.

A new phenomenon is now arising, although only as yet on the horizon, of man's control of evolution and of compressing millions of years of haphazard evolution into possibly a few years of *planned* evolution. This is going to give a much wider meaning to the saying 'Prevention is better than cure', in that unwanted genes may be unmade, and wanted ones made at the molecular level by physicochemical methods. The prospect is even more thrilling than the likely success of man's travels in space. For ensuring man's health, increased useful lifespan and happiness is of far greater importance than his conquest of the Universe.

Is life, despite its enormous complexity, really opening its gates at the biochemical command? There may well be totally unforeseen eventualities. More questions have been raised than answered in the last decade.

The future is promising, but the road is as yet long. In the words of Chargaff in Vienna: 'We have placed a ladder on to the heavens, but a hundred years or more may pass before the first one or two rungs have been established on it'. In the words of Tatum in Moscow, 'I feel that I have the right as a biochemist, and the responsibility as a geneticist and biologist, to warn people that living organisms are much more than mixtures of enzymes and nucleic acid molecules, and that important and basically simple as the molecular aspects of biochemical genetics may seem, Nature may still have many and unpredictable surprises in store for us'.

Another problem may well be posed: Is man by developing new lines of evolution eventually going to oust himself or his likeness from his supreme position in the earth's evolutionary scale? The viruses are considered the lowest organisms in the evolutionary scale, and are genetically RNA controlled. Evolutionary advance led to the development of DNA as the main genetic carrier. Will biochemical genetic research eventually lead to a new and more versatile genetic carrier, say XNA, which would displace man from his forward position in the evolutionary scale?

I doubt whether these questions will be anwered in the VIth Congress, scheduled to take place in New York in 1964, or the VIIth Congress, scheduled to take place in Tokio in 1967.—S.LEWIN.

International Conference on the Physics of Semiconductors

The Institute and Society on behalf of the International Union of Pure and Applied Physics, and the British National Committee for Physics is arranging an International Conference on The Physics of Semiconductors, which will be held at the University of Exeter from 16-20 July, 1962.

Accommodation will be provided in Halls of Residence at the University. Provisional programmes and application forms may be obtained from the Administration Assistant, The Institute of Physics and the Physical Society, 47 Belgrave Square London, S.W.1.

ERRATUM

We regret that an error occurred in the caption to the photograph of Dr. Campbell, on page 694 of the October issue. This should have read P. N. Campbell, D.SC., and not P. S. Campbell, D.SC.

BOOK REVIEWS

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Some 20 pages are devoted to the underlying theory of group separation and these are followed by detailed practical instructions for the analysis of cations. Here the author has introduced a form of nomenclature to describe each stage of the separation. Each group of elements is designated by a roman numeral (from I to VI) and the letters S and L are used to represent solid and liquid phases respectively. If a particular precipitate has been obtained in two stages it is denoted by S^2, if in three stages by S^3 and so on. Similarly a material obtained in various stages as a supernatant is denoted L^2, L^3. To take a familiar example the precipitate of aluminium hydroxide obtained in Group III is denoted III LS which indicates that the original precipitate has passed throuhg a supernatant phase (as sodium aluminate) and has then been reprecipitated (on boiling with ammonium chloride). In this way any precipitate or filtrate obtained during a separation can be identified without doubt, although in some cases the designation becomes a little cumbersome as, for example, when the precipitate of zirconium phosphate obtained in group III is referred to as III S^3L^2S. All things considered, however, this nomenclature represents a praiseworthy attempt to identify all stages of an often complex separation and its use must to some extent alleviate the frustration and annoyance caused by the confusion of unmarked tubes in a full rack. The separation and identification of anions are dealt with in a similar way.

This book is a useful contribution to the available texts on systematic qualitative inorganic analysis and is obviously based on the considerable teaching experience of its author. Many schools and colleges will already have suitable systems of semi-micro qualitative analysis in use but where no satisfactory scheme exists the present volume can be recommended.—C. A. JOHNSON.

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Nevertheless, this indicates the main purpose of the book which is to bring together the known data on 55 uncommon metals, with methods of production from ores and other raw materials, chemical and physical properties, and information on the machining and fabrication of these metals by many varied engineering techniques. The present and potential uses of these metals are also discussed.

As an authoritative and concise reference book, this is a valuable asset to the chemical engineer, the metallurgist, indeed, to anyone concerned with the utilization of these metals either in pure or alloy forms. To the chemist, the lack of information on the compounds of these metals might seem a disadvantage, but this is obviously too large a subject to be included in the terms of reference of this book. Some mention of chemical compounds is made with most of these metals. Perhaps, the most useful aspect to the chemist is the information on the extraction and purification of the metals, which is so often only scantily dealt with in textbooks of inorganic chemistry.

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are available. The editor and publishers are to be congratulated on the high standards maintained by this book which could easily have degenerated into a mass of tabular data, unconnected by any descriptive material.—W. I. STEPHEN.

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The general outlay of the book is good, with a well-proportioned distribution of space between theoretical considerations, technique, methods and results. It is obvious where the author's strength lies, viz. proteins, and this introduces perhaps too strong a bias. The chapters on proteins, particularly the serum proteins, are expertly dealt with. Very good, sound advice is given on staining procedures, photometric measurements and quantitative estimations of protein fractions, based on long and extensive experience, or so it would appear. This definitely should be of great help to the reader in avoiding the pitfalls of inaccurate quantitative evaluation of results both in research and routine work. Interesting information is given on investigations comparing results of stabilized electrophoresis with those of free electrophoresis. All this is accompanied by excellent graphs, tables and illustrations. Sera of pathological cases, both in man and animal, are ably and amply discussed. A rather short chapter on cell and tissue proteins, bigger chapters on haemoglobins, lipoproteins and glycoproteins, and an interesting brief account of the present state of electrophoretic achievements in the field of labelled compounds and isotopes enhance the favourable impression.

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logical screening tests by W. G. Smith. It is of course an almost hopeless task to give anything but a rough sketch of the problems facing the pharmacological aspect of the subject. The author has given a very excellent summary of the present position and recommended a system of planned testing to replace the rather haphazard methods that have been used by some workers. He includes a very interesting analysis of the efficiency and cost of the system recommended.

The second section deals with hypotensive agents by R. Wien. In a relatively short space the author gives a very useful summary of the present position and the search for compounds, particularly of the ganglion-blocking type.

Section three by M. W. Parkes, deals with tranquillizers and here the author is on very difficult ground. In 30 odd pages a very workmanlike account of the present position is given, although some of the clinical aspects of these new compounds are touched upon perhaps a little too lightly.

Section 4 on diuretic drugs, by Prof. Heller and M. Ginsburg, is an excellent summary of the present position and can be read by anyone involved in this field with profit and interest.

Section 5 deals with oral hypoglycaemic drugs by J. D. H. Slater. Here the author has a much more simple subject to deal with than those in the first four. The account is full and very satisfactory.

The final section on antifungal agents by E. P. Taylor and P. F. D'Arcy brings the volume to a close on a high level.

In conclusion it can be stated that the book is attractively produced, is well indexed and contains an excellent series of references and one is very glad to see that these are as up to date as they can possibly be expected to be. It is to be hoped that the succeeding volumes will maintain the standard of this first one.—E. C. DODDS.

pH Measurement and Titration, by G. Mattock with a chapter by D. Ross Taylor, London: Heywood & Co. Ltd. 1961. pp. 406 + xiv, 63s.

pH measurements are used nowadays extensively in various chemical fields, and appreciation of their theoretical and practical aspects is essential. It is therefore not surprising that quite a number of papers, articles and books have been published in this connection. Of the latter the best known are Clarke's 'Determination of Hydrogen Ions', Balliere, Tindall & Cox (1928); Dole's 'The Glass Electrode', Chapman & Hall (1941); Britton's 'Hydrogen Ions', 4th Ed., Chapman & Hall (1955); Ricci's 'Hydrogen Ion Concentration', Princeton University Press (1952); and Bate's 'Electrometric pH Determinations', Chapman & Hall (1954). These books, on the whole, are more concerned with the theoretical aspects than practical ones. Dr. Mattock's book, just published, pays more attention to the practical aspects of pH measurement and brings up to-date the theoretical and practical information in the field.

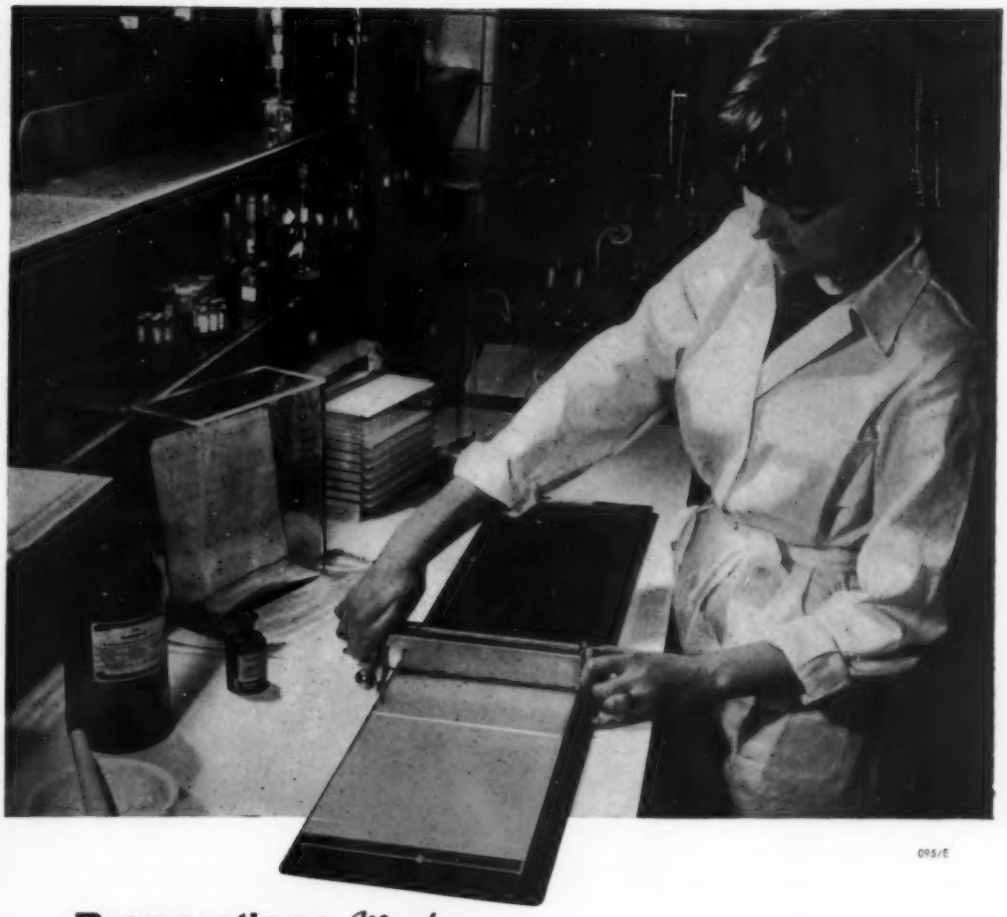
The book is well written and should be easily followed by chemistry graduates and by intelligent undergraduates. It is comprehensive in that it covers the major and most of the minor aspects of pH measurement adequately, and the references given are extensive and cover the field comparatively well. Useful comparison Tables are given, e.g. comparison of alkaline errors for different glass electrodes. The significance of different pH scale standards is gone into in detail.

The increasing use of automatic instrumentation in pH measurements has been met by inclusion of a chapter on Automatic pH Titration. Chapter 10, on pH Measuring Instruments, written by G. Ross Taylor, is indeed a useful chapter, which considers the underlying principles of, and describes and discusses the various instruments on the market. This chapter will be found useful, particularly by persons embarking on extensive pH measurements, in that it gives the various applications and limitations of the many instruments commercially available.

Nevertheless, there are some shortcomings. I was disappointed at the absence of a mention of Michaelis's constant ionic strength buffer (barbitone, KCl, etc.) and of the use of borate-mannitol mixtures as constant ionic strength buffers; of the reactivation of 'poisoned' hydrogen electrodes by boiling in concentrated nitric acid; and with the comparatively short treatment of the use of spectrophotometers in pH measurements, and with the comparatively incomprehensive indexing, for example, the 'cleaning' of glass electrodes is not indexed, though it is found under their 'activation'.

Table 8 (p. 52), giving the 'pH Values of Supplementary Buffers according to the N.B.S. Scale' can result in misunderstanding. It is not stated in the book that the data have been taken from the paper by Manov *et al.* (references 5 and 21 on p. 57 of the book). The impression is gained that these are data which are in *experimental* agreement with those of the N.B.S. standards. This is not the case. The values stated in the 0·05 M borax column deviate significantly from those found experimentally when the N.B.S. buffers are used to standardize a glass electrode. Thus, if one standardizes a glass electrode with 0·01 M borax at 20°C. at pH 9·22 and then measures the pH of a 0·05 M borax, the pH measured is 9·18—and not 9·25 as given in the Table, the value being a theoretical value deduced from various considerations including activity coefficients; the difference is indicative of the difficulties encountered in correlation of pH scales. Parallel remarks apply to other pH values given under the 0·05 M borax column; the experimental pH values are lower than those of the 0·01 M borax pH values, and not higher.

I would recommend this book not only for the average chemistry laboratory but also to members of research laboratories in which pH measurements are carried out extensively, and particularly to the young graduate about to undertake research on problems which involve appreciation of pH values.—S. LEWIN.



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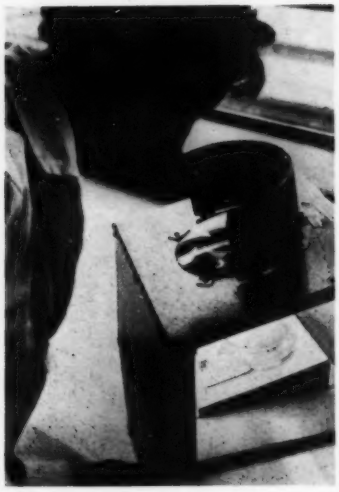
LABORATORY EQUIPMENT & APPLIANCES

Optical Viewing Panel

An optical viewing panel was recently constructed on behalf of the Sheffield Corporation Waterworks.

This equipment is being used to count the bacterial colonies developing



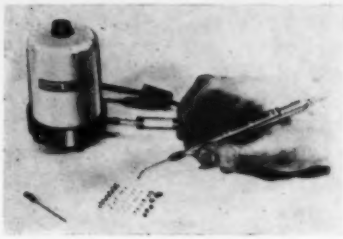
Optical viewing panel.

in samples of water incubated on agar jelly in glass Petri dishes. This is a technique commonly employed in medical, water, dairy and similar laboratories and is generally referred to as the 'bacterial plate count on nutrient agar'. The particular advantages afforded by the instrument lie in the method of supporting and illuminating the Petri dish, and in the built-in magnifier to ensure accurate counting.

(Manufacturers: Optical-Mechanical (Instruments) Ltd., 36a, Station Road, Egham, Surrey).

Vacuum Tweezer System

A new vacuum tweezer system, V-100, which is portable and self-contained, operates from a.c. mains supply and consists of a patented electro-magnetic vacuum generator and a vacuum pencil with stainless steel pick-up tip. No larger than a tea cup, the generator is provided with an adjustable pressure control. The vacuum pencil is similar in size to a propelling pencil, and is connected to the generator by flexible tubing which incorporates a dirt filter.



Vacuum tweezer system.

Various shapes and sizes of pick-up tip are provided.

Operation is easier, and less likely to damage the component being picked up, it is claimed, than any mechanical handling arrangement. To hold a component the operator simply touches its surface with the pick-up tip of the pencil, at the same time covering a small opening in the pencil body to maintain vacuum pressure. Uncovering the opening will release the pressure and the component.

The system ensures fast handling of even the most delicate parts without damage, and is suitable for laboratory use.

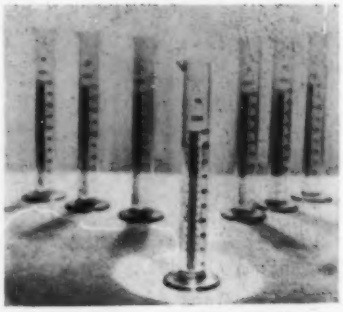
(Sole Concessionaires in the U.K.: L. E. Simmonds Ltd., 5 Byron Road, Harrow, Middlesex).

Acrylonitrile Measuring Cylinders

A prolonged survey of the shortcomings and the failures of plastic materials used in the manufacture of laboratory ware has revealed that above all else it is the lack of transparency and clarity that has created the bulk of criticism of these materials. Every material has its limitations and no single one has yet solved the problems entailed completely.

In an attempt to overcome these problems and to provide a range of laboratory equipment which can be used effectively, investigation was carried out as to the most suitable material which might be used for this purpose. As a direct result of this investigation a 'glass clear' 100 ml. measuring cylinder moulded in Acrylonitrile has been introduced. This is the first of four sizes (10, 25, 50 and 100 ml. capacity) which will also be followed by other sizes and by other items of laboratory ware which have hitherto been available only in materials which are subject to the disadvantages of limited or no transparency.

Tyril 767, which is the material used for these mouldings, is a rigid thermoplastic copolymer of styrene and acrylonitrile. It has good heat resistance, toughness and is resistant to most chemicals including many aliphatic hydrocarbons and to petrols except those having high aromatic content. It is also resistant to many other commonly used chemicals and solvents including alkalis, salts, dilute mineral acids and lower alcohols. This material is particularly suitable for applications involving direct contact with food products.



Acrylonitrile measuring cylinders.

Further development work is being carried out with mouldings and extrusions of this material and of others in order to improve the quality and effectiveness when applied to the exacting requirements of science, industry and the laboratory. Already available are a matching range of 12 sizes of beakers, from 10 ml. to 5 litre, moulded in both Polypropylene and high density Polythene, Polythene aspirators from 2½ litre to 60 litre capacity and these are fitted with a non-leak, non-drip tap, PTFE and Polythene coated stirring magnets, pipette washers and effective Polythene syphons together with a variety of other plastic laboratory equipment and fabrications.

(Manufacturers: Technical Treatments Ltd., Russell Laboratories, Rye Lane, Otford, Kent).

Pulse Polarograph

Following his work on square-wave polarography, Dr. G. C. Barker of Harwell has developed a pulse technique which gives greater sensitivity to irreversibly reduced ions, greater independence of base electrolyte impurities, and polarograms of greater certainty and ease of measurement.

The Southern-Harwell Pulse Polarograph provides both normal and derivative polarograms, and is believed to be the most sensitive polarograph available.

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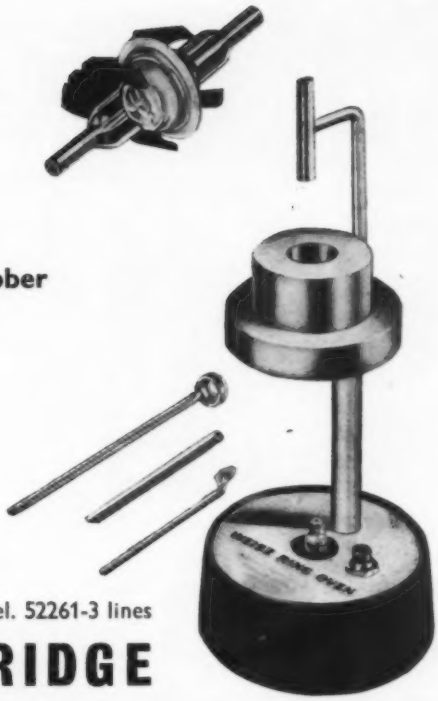

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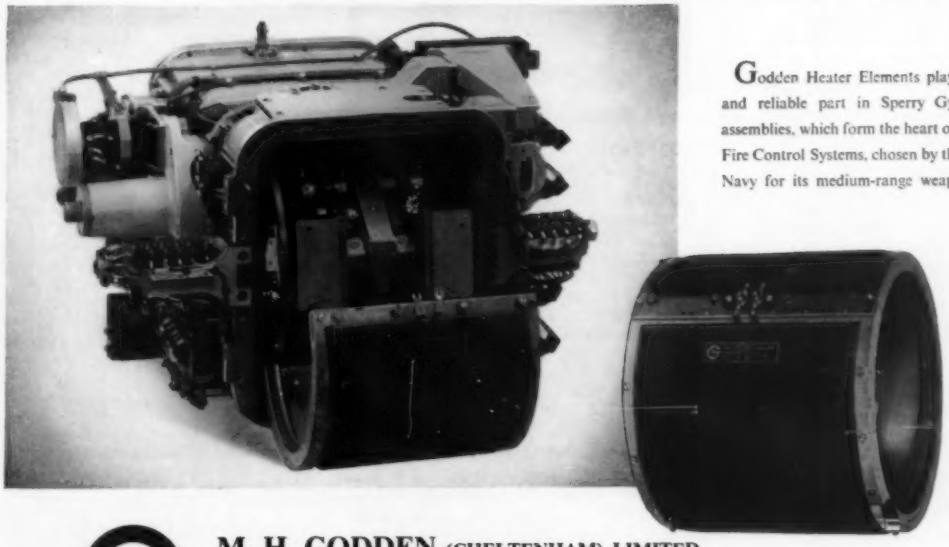

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Pulse polarograph.

(Manufacturers: Southern Analytical Limited, Frimley Road, Camberley, Surrey).

Automatic Sample Counting Assembly

This equipment has been designed to count up to 50 solid radioactive samples and to print out on 2½ in. wide paper tape the sample number, the recorded count and the elapsed time. The sample changer (I.D.L. type 2014) is based on a U.K.A.E.A. design and uses a patented system of spiral cams to move the samples into position. It is used in conjunction with an I.D.L. 1700 scaler, a programme and read out unit and a slightly modified Addo-X special digital printer.

Any number from 1 to 50 samples, prepared on either 1 in. or 2 in diameter flat aluminium planchets, may be arranged to be offered in turn to a thin end window G.M. tube (1 in or 2 in. dia.). Lead shielding is provided around the counter to reduce the background from external radiation and from the adjacent samples. The planchets are loaded into moulded plastic trays, constructed from a material with a low coefficient of friction, and the trays are arranged in a vertical stack at one end of the sample changer. The mechanism takes the tray from the bottom of the stack and moves it in a horizontal direction into a position under the counter. A micro-switch senses the tray and, when it is correctly positioned, switches off the drive motor and initiates a counting cycle.

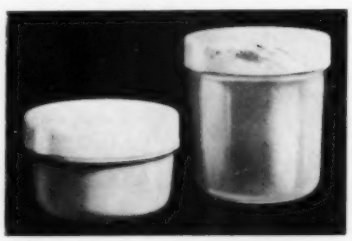
At the conclusion of a count, which is terminated by the preset count or preset time circuits in the scaler, whichever is first reached, the print-out sequence is started and a fresh sample is moved into position; the samples already counted are stacked vertically on the other side of the sample changer to the original stack. When the print-out is complete and a new sample in position the complete counting sequence is repeated.

The programme can be arranged so that when all samples have been counted once (and are then stacked in the reverse order) the whole equipment is switched off or two other alternatives are available. The samples may be re-stacked in their original order and the equipment switched off or they may be re-stacked and then re-counted in their original order. This latter sequence may be automatically repeated as often as desired.

(Manufacturers: Isotope Developments Ltd., Bath Road, Beenham, Reading, Berks.)

New 2 and 4 oz. Polypropylene Jars

A complete new line has been introduced in the form of a 2 oz. and 4 oz. injection moulded polypropylene jar, as well as in polystyrene. Supplied complete with screw on cap, each size is, as the illustration shows, both practical and pleasant to the eye.



New injection moulded jars.

It is impermeable, it is resistant to heat or chemical attack and can be subjected to sterilization by heat or radiation either before or after the filling operation. It can be used for the packaging or storage of most chemically reactive liquids, solids and semi-solids. It is light in weight and, under all reasonable conditions, unbreakable, thus showing considerable savings in storage space, packing, postage, transport costs and losses through breakage.

(Manufacturers: Dines Plastics Ltd., Whitehall Lane, Grays, Essex.)

Curve Follower

The curve follower enables the Bryans 'X' and 'Y' co-ordinate plotting table to be used to read-off from a curve placed on the table, and produce a voltage proportional to the 'Y' co-ordinate of the curve when the appropriate 'X' co-ordinate is fed into the table. This is very useful for instance in numerous analogue simulations where some non-linear characteristics have to be introduced, or for linearizing a non-linear input as required by a thermocouple or square root law, etc.

A curve drawn in conducting ink or wire is fed by alternating current from the curve follower attachment. The created magnetic field around the conducting curve is sensed by an inductive head which feeds any error signal into the transistor amplifier. The D.C. voltage output from this amplifier is fed to the 'Y' axis servo-loop to maintain the sensing head in position over the curve. The sensing head must be initially placed approximately over the curve using the shift controls of the 'X' and 'Y' co-ordinate plotting table. The 'X' co-ordinate input is applied using the normal 'X' co-ordinate controls. The 'Y' co-ordinate output voltage is brought out, together with an opposite polarity voltage from the pen shift control for zero suppression, to the signal plug which is connected to appropriate equipment according to requirements, e.g. analogue simulator.

(Manufacturers: Bryans Aeroquipment Ltd., 1 and 15 Willow Lane, Mitcham, Surrey.)

Hy-Flo Pumps

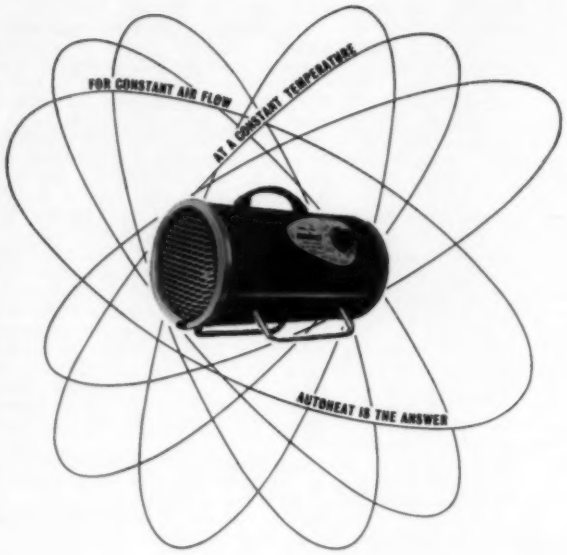
Although originally intended for the aquarist, Hy-Flo pumps now find a wide range of uses in hospitals, laboratories, universities, schools and industry and for animated display units. They are also incorporated in the Coulter automatic blood cell counter and cell size analyser. The latest development in the use of these pumps is in conjunction with incubators for the very exacting scientific devices to save the lives of premature babies.

These pumps are silent in operation, self-starting (shaded pole induction type motor), suitable for continuous operation, without interference to radio or television, moderate in electricity consumption, have slow running piston pumps, are not subject to the annoyance of water syphoning back accidentally, able to aerate successfully in water from 4 in. to 4 ft. in depth, fitted with precision ground pistons and valve block faces and hardened precision ground crank pins and spindle, supplied with main bearing of phosphor bronze, fitted with wick lubrication to all moving parts and provided with earth connection made via approximately 6 ft. of three-core cable.

(Manufacturers: Medcalf Brothers, Cranbourne Road, Potters Bar, Middlesex).

New Developments in Flame Photometry

Many improvements have been incorporated in the Carl Zeiss flame photometer, Type P.F.5, an instrument which enjoys extensive use in science and industry. These improvements will



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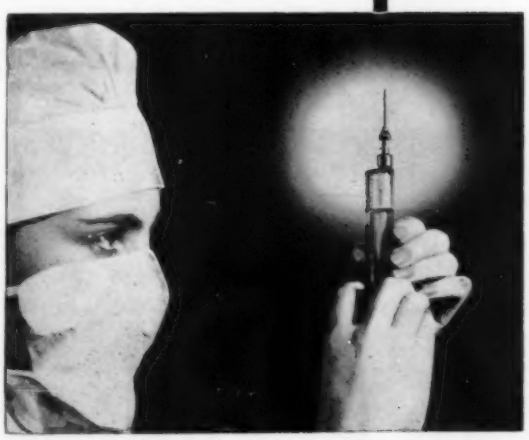

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be included in all instruments supplied after September 1:

1. Compensating voltage has been doubled.
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3. As an additional safety precaution, a quick-change coupling for the fuel gas supply has been incorporated at the rear of the instrument.
4. The newly-designed atomizing chamber incorporates a filament inset which ensures better separation of fine drops, thereby reducing the disturbance in emission of the analytical element by third components, i.e. phosphate and sulphate.
5. A new glass burner includes an inter-changeable nozzle made of Trovidur. This gives an improved mixture of gases so that the flame burns with complete steadiness.

(U.K. Agents for Carl Zeiss: Degenhardt & Co. Ltd., 6 Cavendish Square, London, W.1).

Process Differential Refractometer

Control of liquid process streams based on very small changes in index of refraction of the stream compared to a sealed reference standard or a flowing reference standard has been made possible by a new process differential refractometer offered by Waters Associates, Framingham, Massachusetts.

Process differential refractometer.

The new refractometer can measure full scale changes in refractive index as small as 0·00005 with a stability better than 1 per cent of full scale in 24 hours. The sensitivity of the new instrument is 0·5 per cent of full scale, or 0·00000025 R.I.

A unique and important feature available in this refractometer is the flowing reference cell. With this design, true, accurate blending of two streams can be accomplished even though one stream varies in composition. Accurate blending control was impossible in previous refractometers using a sealed reference standard where the composition of one stream would change thus changing the refractive index. In the flowing reference cell design one stream flows through the reference cell continuously and the same stream plus the addition of the blended material flows through the sample cell.

The second important feature, the extremely high sensitivity of the apparatus now makes it possible to measure p.p.m. impurities in final product streams. The full scale sensitivity is 10 times greater than most conventional refractometers. Ranges as narrow as 0 to 500 p.p.m. full scale can be provided and when the sealed cell design is used an ultra pure standard can be sealed permanently in the reference cell.

In operation, the refractometer measures the amount a light beam is bent when the refractive index of a liquid sample changes. A light beam from an incandescent lamp is directed through a slit, mask and lens to the sample cell through which the liquid sample is flowing. The beam passes through the sample liquid and the reference standard to a mirror mounted behind the sample cell. The mirror reflects the beam back through the cell for a second traversal of the sample liquid and the reference standard.

At this point, the location of the light beam depends upon the relative refractive indexes of the sample and the reference standard. The beam is then divided by a beam splitter mirror. Each half of the beam falls on a cadmium sulphide photocell. The photocells form two arms of a Wheatstone bridge. If the sample changes, the beam is refracted more or less and the amount of light reaching one photocell changes with respect to the other photocell.

The bridge circuit becomes unbalanced and the resultant signal is amplified causing the balancing motor to drive. The beam deflector plate is geared to the motor and turns in a direction to equalize the light falling on the two photocells. The more the refractive index of the sample deviates from the reference the greater the unbalance in the bridge circuit and the more the deflector must turn to rebalance the split beams.

The position of the deflector, therefore, is a measure of the refractive index of the sample. At the same time the motor drives the deflector, it drives a helipot across which an electrical voltage has been impressed. The output signal from the helipot activates the meter and a remote recorder which indicates directly the concentration of the measured variable in the liquid sample.

The refractometer is available either in explosion-proof or non-explosion-proof housings.

(All enquiries to: Scientific Furnishings Ltd., Poynton, Cheshire).

Carbon Monoxide Detector-Plate

Carbon monoxide is a particularly treacherous hazard; it cannot be seen, smelt or tasted, even when present in lethal concentrations. It occurs wherever there is a combustion of organic matter. 0·3 per cent carbon monoxide in inhaled air is sufficient to cause immediate loss of consciousness and death within half an hour.

The presence of carbon monoxide has hitherto been detected by various monitoring instruments operating on

The new detector monitor, introduced in Denmark, consists of a small plastic plate with disc-like insert containing a special sensing substance, the active principle of which is Palladium chloride suspended in a silicagel of the same yellow tint as the plate.

IS THE NEW

Apart from its appearance, which we venture to think is considerably improved, there are no doors to open, the Nessleriser glasses can be more easily inserted and removed, and the design gives built-in provision for blue glass filters for use with white light attachments. Most metal parts have been eliminated. The range of interchangeable discs for use with the instrument provides permanent colour standards for some thirty analytical procedures, in addition to seventeen discs for pH determinations. The price, complete with any nine-standard disc and a pair of matched Nessleriser glasses, is £12.10s.0d.

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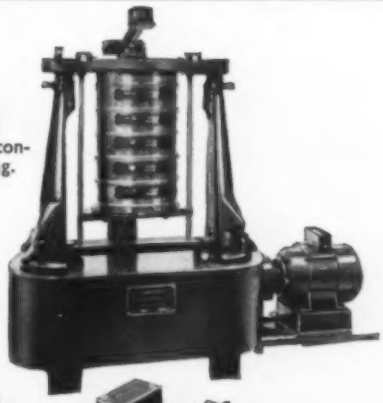

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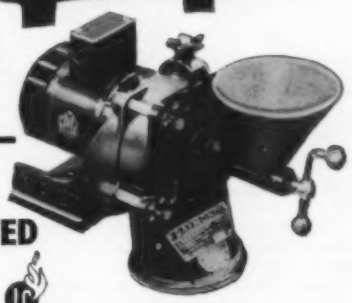

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air-sampling principle. This means that protection is limited to the sampling period.

A new detector monitor has recently been introduced in Denmark. It consists of a small 5 × 5 cm. plastic plate with a disc-like insert containing a special sensing substance, the active principle of which is Palladium chloride suspended in a silicagel of the same yellow tint as the plate. In the presence of a carbon monoxide the Palladium-salt is reduced and the insert turns grey or black, depending on the degree of concentration of the gas. Carbon monoxide in a concentration of 0·01 per cent takes less than 15 minutes to detect, five minutes if the concentration is 0·02 per cent. Detection is practically immediate when this proportion reaches 0·05 per cent.

After blackening by influence of carbon monoxide, self-regeneration will take place by oxygenation through exposure to non-contaminated air for 20 minutes to two to three hours. The plate can be used several times before losing its efficiency. Blackening after exposure to impurities of the air (hydrogen sulphide), involves loss of efficiency.
(Sole Distributors in U.K.: Herzbi Ltd., 57 Lordship Park, London N.16.)

CATALOGUES, BROCHURES & LEAFLETS

Received from Manufacturers

The Diamond Research Laboratory.—A booklet has been prepared by the Industrial Diamond Information Bureau of 2 Charterhouse Street, London, E.C.1, which describes the fundamental and applied research into the diamond undertaken by the Research Laboratory in South Africa and to acquaint users of industrial diamonds with the service the laboratory provides.

Viscometers.—Poulten, Selfe & Lee Ltd., of Wickford, Essex have issued a brochure giving details of their glass capillary viscometers and falling-sphere tubes as follows: U-tube, miniature U-tube, suspended-level, suspended-level (shortened form), suspended-level (miniature), Cannon-Fenske, U-tube reversed flow and falling sphere tubes.

Flexible Waveguides.—The latest specifications for the range of flexible waveguides in WG16 from 1·12 to 40·00 kMc/s. are contained in the new Flexaguide catalogue issued by W. H. Sanders (Electronics) Ltd., Gunnels Wood Road, Stevenage, Herts.

MEETINGS FOR THE MONTH

Mid-December to Mid-January

We give below a selection of meetings of interest to laboratory workers. Details are as full as possible at the time of going to press, but readers requiring further information or confirmation of arrangements should contact the organizers.

December 11

The Chemical Society (Durham). Joint meeting with the Durham Colleges Chemical Society. At the Science Laboratories, The University. 5 p.m. 'Seeing Molecules with Microwaves', by Dr. J. Sheridan, M.A.

December 12

The Society of Instrument Technology (Chester Section). At Stanley Palace, Watergate Street, Chester. 7 p.m. 'Design, Application and Selection of Automatic Control Valves', by P. Stone.

December 13

The Society for Analytical Chemistry (Midlands Section). At The University, Birmingham, 15. 7 p.m. 'Fluorescent Indicators for Metals', by Dr. W. I. Stephen.

The Society of Instrument Technology (Control Section). At Manson House, 26 Portland Place, London, W.1. 6.30 p.m. for 7 p.m. 'Application of Complex Plane Methods to System Design', by P. K. M'Pherson, M.Sc., A.M.I.Mech.E.

The Society of Instrument Technology (Manchester Section). At the Literary and Philosophical Society, 36 George Street, Manchester, 1. 6.45 p.m. 'Instrumentation for Oil Fired Glass Furnaces', by N. I. Walker.

December 14

The Chemical Society. In the Lecture Theatre, The Royal Institution, Albemarle Street, London, W.1. 7.30 p.m. Liversidge Lecture, 'Stereospecific Polymerization', by Professor C. E. H. Bawn, C.B.E., Ph.D., F.R.S

The Society for Analytical Chemistry (Biological Methods Group). At The Feathers, Tudor Street, London, E.C.4. 6.30 p.m. Annual General Meeting followed by a Discussion Meeting on 'Assessment of Antiatherosclerotics.'

The Society of Instrument Technology (Bristol Section). The Department of Physics, University of Bristol, The Royal Fort, Bristol, 8. 7.30 p.m. 'The Atomic Clock', by Dr. L. Essen, O.B.E., F.R.S., D.Sc., A.M.I.E.E., F.B.H.I.

The Society of Instrument Technology (Liverpool Section). At the Merseyside and North Wales Electricity Board Industrial Centre, Paradise Street, Liverpool. 7 p.m. 'The Versatility of the Electronic Potentiometer', by F. W. J. Howard.

December 21

The Society of Instrument Technology (Grangemouth Section). At the Leapark Hotel, Bo'ness Road, Grangemouth. 7 p.m. 'Instrumentation of Space Vehicles', by Dr. A. E. Roy B.Sc., Ph.D., F.R.Ae.S.

1962

January 3

The Society of Instrument Technology (South Yorkshire Section). At Brown-Firth Research Laboratories, Princess Street, Sheffield. 4. 'Film Night'.

January 5

The Society of Instrument Technology (Fawley Section). In Room 4a, Administration Building, Esso Petroleum Co. Ltd., Fawley. 5.45 p.m. 'Measurement of Flow', by J. Singer, A.M.I.Mech.E.

B.D.H. Leaflets.—New entries in the catalogue of British Drug Houses Ltd., of Poole, Dorset are listed in the August 1961 issue of their monthly leaflet as follows: *Organic and Inorganic Chemicals*—Furan; high purity metals; l-phenyl-tetrazoline-5-thione sodium salt; tetra-*n*-propyl-ammonium iodide. *Biochemicals*—sodium pyruvate. The September issue contains the following: *Organic and Inorganic Chemicals*—Glass beads for gas chromatography; *n*-heptylamine. *Amino Acids*—*N*-acetyl-L-glutamic acid; glycine anhydride; *N*-phthaloyl-DL-α-alanine. *The Lovibond Comparator*—new Lovibond comparator discs.

Materials for Gas Chromatography.—May & Baker Ltd., of Dagenham, Essex, have published the second edition of their booklet 'Materials for Gas Chromatography.' An innovation is the introduction of the first members of a new range of specialized stationary phases which are currently attracting attention in the literature. These materials are carefully purified but not, as yet, standardized; if any should become of sufficient importance in the future, then they will be transferred to the Embaphase range. The range of supporting media has been extended by providing Embacel in additional size grades and brick dust has been added for the benefit of those who still prefer to use it instead of kieselguhr. The tabulation of stationary phases by name and by application has been much enlarged and a new appendix tabulates correction factors for pressure drop across the gas chromatography column.

NEWS

Cambridge Instrument Company's New Instrument Assembly Wing

The Cambridge Instrument Company's new instrument assembly wing, officially opened by the Minister of State at the Board of Trade, Sir Keith Joseph Bt., M.P., at Cambridge on 23 October, marks another stage in an expansion programme designed to meet the increasing demand for measuring instruments in science and industry. It represents an additional 15,000 sq. ft. of production space and has already begun to play its part in enabling the company to exploit more fully—in practical manufacturing terms — the growing potential offered by the intensive research and development work now in progress.

An example of this is the Microscan x-ray analyser, whose rate of production is expected to double itself within the next few months. This instrument, which is used for investigating the microstructure of metals and minerals, was partly responsible for the record export turnover achieved in 1960, which was 47 per cent higher than in 1959.

Other recent events have been the doubling, in May 1959, of head office accommodation, the opening of new research laboratories at Cambridge in October of the same year, the addition of an extension and a staff clubhouse at the Muswell Hill (London) factory, in January and May 1960, and the purchase in June 1960, of the whole of the ordinary share capital of Electronic Instruments Limited — designers and manufacturers of high quality measuring units for the scientific, industrial and nuclear instrument fields.

Outstanding features of the new wing are the clean functional sweep of its two raised storeys, the ground-level car park beneath, and the harmonious way it blends with the research laboratory building—opened two years ago—to which it has been planned as a logical architectural extension. The two raised storeys are joined at one end to the research laboratories and at the other, by means of a two-storey link block, to existing works buildings.

The addition of a new instrument assembly wing to the Research Laboratories of the Cambridge Instrument Company provides a pleasing frontage to the Chesterton Road and River Cam. (*Photo: Henk Snoek*).

News from the U.S.A.

Chemists' Salaries

A United States Government survey, summarized in *Chemical and Engineering News*, shows that chemists' salaries, at comparable levels, are slightly below those of engineers, mathematicians and accountants, and very considerably below those of attorneys (lawyers). They still represent a very satisfactory standard compared with British levels.

Six grades of chemists are quoted, and the most junior, an inexperienced beginner with a bachelor's degree, receives a median salary of 113 dollars per week (£38). At the stage where his specialized knowledge enables him to develop details of an assignment he receives about 155 dollars per week (£55). The senior ranks, in charge of staff and programmes should expect 215 and 250 dollars respectively per week (£77 and £89). A research and development director has a median of 350 dollars per week (£125).

Comparing these figures with the B.A.C. scale, it will be seen that a beginner in the U.S.A., say at 23, receives 2·7 times the salary he would expect in Great Britain. A senior qualified chemist without special responsibility would receive slightly less than double his B.A.C. rating at age 35. Chemists who attained executive level in the U.S.A. again find a sharp increase in ratio to about 2·3 of that prevailing here, whilst the median for research directors is again 2·6 of the B.A.C. minimum for posts of exceptional responsibility.

If a figure of about 1¾ is taken as the cost of living ratio between the two countries (see *British Chemist*, November, 1960, p. 61), it will be seen that the young chemist, and the chemist with executive responsibility are considerably better off, but that the man without special responsibility in middle life has only a very slightly higher standard of living.

Mr. B. J. Etherington

Mr. B. J. Etherington has been appointed Marketing Manager to H. J. Elliott Ltd.

For the past eight years Mr. Etherington has been associated with the laboratory and scientific industry in his capacity of Export Manager to Electrothermal Engineering Ltd. He is a member of the Institute of Marketing and Sales Management, and sits on the Export Committee of the Scientific Instrument Manufacturers' Association.

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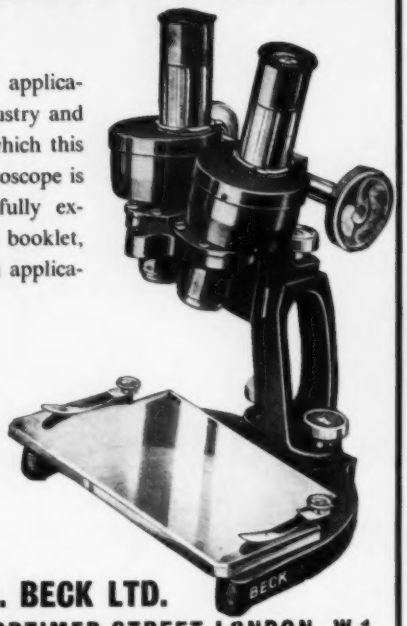

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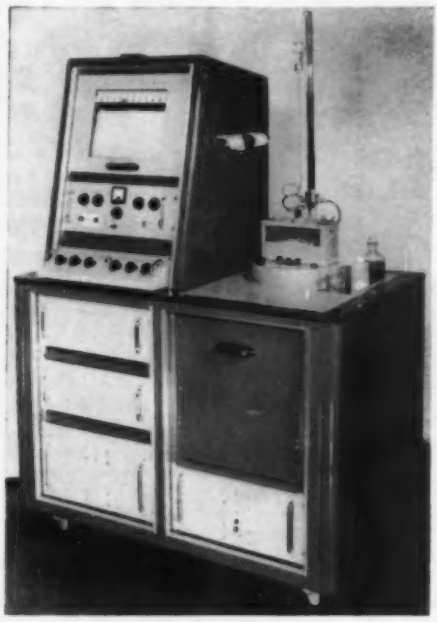

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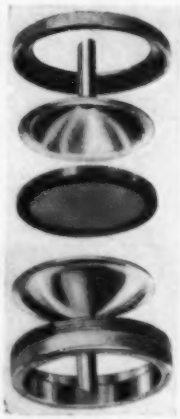

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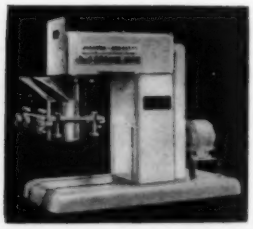

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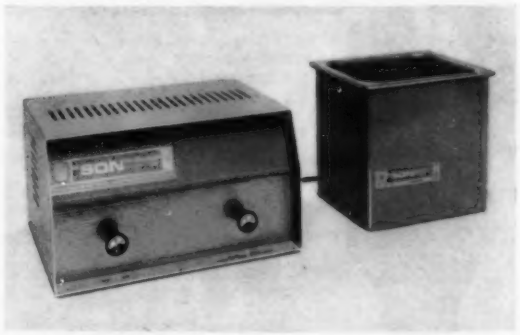

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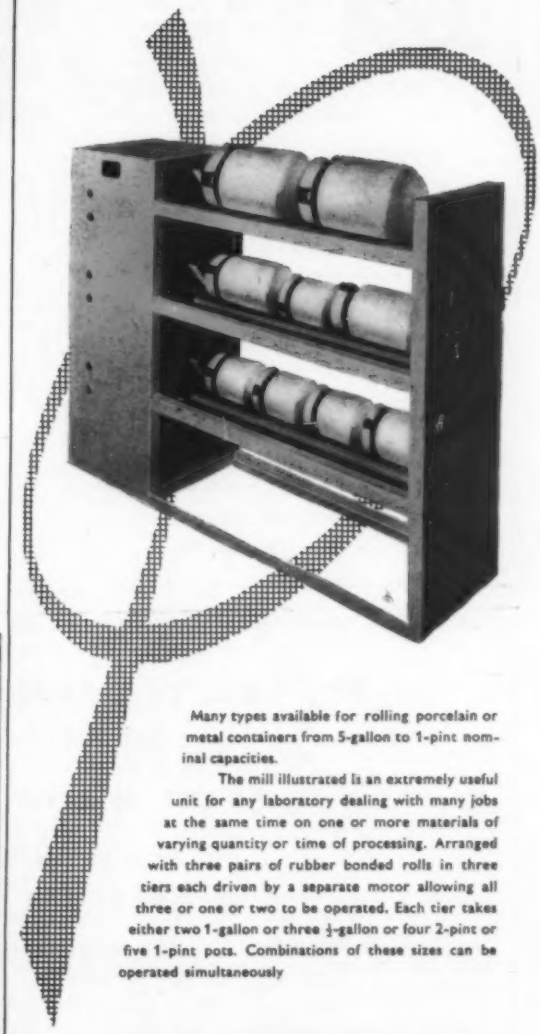

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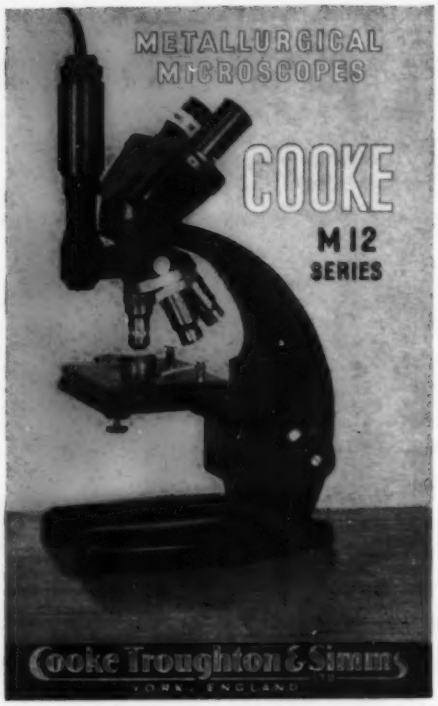

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Index to Advertisers

Adelphi Manufacturing Co. Ltd. 926
Aimer Products Ltd. 929
Anderman & Co. Ltd. 914
Associated Electrical Industries Ltd. ... ... 849 & 859
Astell Laboratory Service Co. Ltd. 852
Austen Pumps Ltd., Charles ... ... November
Baird & Tatlock (London) Ltd. ... ... Cover i
Baker, C., Instruments Ltd. 852
Barcham Green, J., Ltd. 914
Bausch & Lomb Optical Co. Ltd. 926
Beck, R. & J., Ltd. 914
Beecroft & Partners Ltd. 850
Bendix Ericsson U.K. Ltd. ... ... November
Boro' Laboratories & Appliance Co. Ltd. 930
British Drug Houses Ltd., The 911
British Electrical Resistance Co. Ltd. 858
Brown (Brownall) Ltd. ... ... November
Camlab (Glass) Ltd. 907
Classified Advertisements ... ... 931 & 932
Cooke, Troughton & Simms Ltd. 930
Copley, F., & Co. 927
Coulter Electronics Ltd. ... ... November
Davey & Moore Ltd. 856
Degenhardt, A. H., & Co. Ltd. 854
Difco Laboratories, Inc. .. .. Cover iv
Distillers Co. Ltd. The 846
Edina Ltd. (Emoco) 909
Edwards High Vacuum Ltd. ... ... November
Electrothermal Eng. Ltd. ... ... October
Elga Products Ltd. ... ... November
English Clock Systems ... ... November
Esco (Rubber) Ltd. 855
Evans Electroselenium, Ltd. ... ... October
Flaig, W. G., Ltd. ... ... November
Gallenkamp, A., & Co. 844
Genevac Ltd. 853
Gerrard, T., & Co. Ltd. 930
Godden, M. H. (Cheltenham) Ltd. 907
Gurr, Edward Ltd. 930
Gurr, George T., Ltd. 931
Hawkesley & Sons Ltd. 929
Headland Eng. Developments Ltd. 926
Hearson, Charles, & Co. Ltd. 860
Hedin Limited ... ... November
Hone, F. J., & Co. Ltd. 925
Hopkin & Williams Ltd. ... ... Cover ii
Hudes Merchandising Corporation Ltd. 848
International Combustion Products Ltd. 911
Isopad Ltd. 847
Jackson Crockatt, J. G., Ltd. 926
Jencons (Scientific) Ltd. ... ... November
Jobling, James A., & Co. Ltd. ... ... 842 & 843
Lock, A. M., & Co. Ltd. 860
Mallory Batteries Ltd. 841
May & Baker Ltd. ... ... November
Merck, E., A.G. 905
Moncrieff, John, Ltd. ... ... Cover iii
Newton Maine, B., Ltd. 930
North of England School Furnishing Co. Ltd. ... 848
Orme Scientific Ltd. 846
Oxo Limited (Oxoid Division) ... ... 845 & 857
Pascall Eng. Co. Ltd. ... ... 854 & 929
Perry, Charles, & Co. Ltd. ... ... November
Poulten Selfe & Lee Ltd. ... ... November
Purkis, Williams, Ltd. 931
Quickfit & Quartz Ltd. ... ... November
Reeve, Angel H., Co. Ltd. ... ... November
Scientific Industries International Inc. (U.K.) Ltd. ... 844
Scorah, L. V. D., M.Sc. 927
Silverson Machines Ltd. 858
Southern Analytical Ltd. 925
Spencer Components 932
Stanton Instruments Ltd. ... ... November
Surgical Equipment Supplies Ltd. 909
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Thermal Syndicate Ltd., The 851
Townson & Mercer Ltd. 862
United Trade Press Exhibitions Ltd. 928
Watson, W., & Sons Ltd. 861
Wild-Barfield Electrical Furnaces Ltd. 856
Zeal, G. H., Ltd. ... ... November

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continued from page 932

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OFFICIAL APPOINTMENTS

LABORATORY TECHNICIAN required by the Bechuanaland Government Veterinary Services Department either on probation for 3 years for admission to the pensionable establishment or on contract for a tour of 3 years with gratuity at rate of 12½ per cent of total emoluments drawn.

Commencing salary up to maximum according to experience in scale (including Inducement Pay) equivalent to £848 rising to £1,488 a year, plus an allowance of £60 a year for married officers or £30 a year single officers. Education allowances up to £288 a year. Liberal leave on full salary. Free passages.

Candidates must be F.I.M.L.T. and will be employed on technical duties at a Veterinary Research Laboratory on pathological and bacteriological work. Apply to Crown Agents, 4 Millbank, London, S.W.1 for application form and further particulars, stating age, name, brief details of qualifications and experience and quoting reference M3C/53675/LAQ.

SITUATIONS VACANT

LABORATORY TECHNICIAN with 'A' levels in Chemistry and Physics or equivalent qualification, preferably with experience, required for Biochemical Department for analytical and gas chromatographic work on lipids. The salary will be dependent on qualifications and experience. Apply to the Assistant Secretary, The Lister Institute of Preventive Medicine, Chelsea Bridge Road, London, S.W.1.

SURREY EDUCATION COMmittee. Laboratory Technician required in Department of Chemistry for servicing and maintenance of chemical and physical equipment, glass blowing and construction of apparatus. Experience in electronics and/or spectroscopy an asset. Qualifications, C. & G. Laboratory Technicians certificate, O.N.C. Chemistry, or minimum of four years appropriate practical experience. Salary, Misc. III £555-£625 p.a. or Misc. IV £625-£685, plus London Allowance £25-£40. Overtime payable in excess of 38 hours per week worked. N.J.S. Conditions of Service, including superannuation. Applications to Principal, Kingston Technical College, Fassett Road, Kingston-upon-Thames.

PHYSICIST REQUIRED, AGE 25-35, with good Degree and some experience in electrical discharge phenomena in gases for studies in this field and design and development of corresponding equipment. 5-day week. Please write, quoting No. 36, to Personnel Officer, The British Oxygen Co. Ltd., Scientific Division, Deer Park Road, London, S.W.19.

University of Melbourne

Technical Officer required by Biochemistry Department for organization at technical level of undergraduate practical classes. The successful applicant will preferably have had experience in a teaching laboratory and will be required to take up his appointment in Melbourne on or about 1 March, 1962.

Commencing salary, according to qualifications and experience, within the range £A1,210—£A1,287 or £A1,287-£A1,376 with opportunities for promotion.

A candidate with exceptional qualifications could be considered for initial appointment at a higher level.

Half salary will be paid from date of embarkation to arrival. Assisted passage will be arranged.

Applications should reach the Secretary, Association of Universities of the British Commonwealth (Branch Office), Marlborough House, Pall Mall, London, S.W.1, by **18 December, 1961.**

University of Adelaide.
Technical Officer, Senior Technical Assistant or Technical Assistant

Applications are invited for the abovementioned position in the Department of Botany.

Salary Scales:

Technical Officer: Male: £A1,416-45-1776, with a bar at £A1,596.
Female: £A1,063-33⅓-1333, with a bar at £A1,198.

Senior Technical Assistant: Male: £A1,335-35-£A1,580.
Female: £A1,003-26-£A1,185.

Technical Assistant: Male: £A1,126-35- £A1,336.
Female: £A846-26-£A1,002.

Qualifications: Considerable laboratory experience and competence with apparatus and techniques in biology, and preferably with the preparation and collection of botanical teaching materials and histological methods.

Duties: Supervision of basic laboratory apparatus and preparation of undergraduate classes.

General: (a) No provision is made for travelling expenses, but arrangements could be made to help a candidate from the United Kingdom to secure an assisted passage (and passages for his wife and family also) under the Commonwealth Migration Scheme.

(b) Preference will be given to a qualified male applicant, but women also are invited to apply.

(c) Any further information desired will be supplied on request to the Registrar.

Applications, giving particulars of qualifications and experience, marital status, the names and addresses of two referees, and the time when the applicant would be free to assume duty, should be sent to the Registrar, University of Adelaide, Adelaide, South Australia, **not later than 20 December, 1961.**

COURSES

Norwood Technical College, Knight's Hill, London, S.E.27

Principal: H. V. Hillman, B.Sc., M.A., F.R.I.C.

Short Course on an introduction to Micro- and Semi-Microchemical Methods

Commencing 13 January 1962 a course of 12 lectures and appropriate practical work designed to survey the principal branches of Chemistry in which small-scale methods have been successfully applied, will be held on Saturday mornings from 9.15 a.m. to 12.30 p.m. Application forms for admittance to the course and detailed syllabus of the lectures and practical work may be obtained from the Secretary of the College. Fee for the course is £1.

CLASSIFIED ADVERTISEMENTS
continued on page 931

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is a highly selective medium for isolation of *Salmonella typhosa*. The unusual selective properties of this medium permit the use of large inocula of faeces and other suspected material without overgrowth of extraneous intestinal bacteria.

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is an excellent differential medium for use in conjunction with Bacto-S S Agar and Bacto-Bismuth Sulphite Agar. This medium supports rapid and luxuriant growth of even the most fastidious strains of the typhoid-dysentery group. Although MacConkey Agar does not inhibit coliform bacteria it does afford excellent differentiation of colonies of pathogens from those of the lactose fermenting bacilli.

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